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SEMI-ANNUAL PROGRESS REPORT
DETERMINATION OF THE EMISSIVITY OF MATERIALS

by
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prepared for
National Aeronautics and Space Administration
Contract NAS3-4174

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

EAST HARTFORD • CONNECTICUT

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Semi-Annual Progress Report
Determination of the Emissivity of Materials

Report Period: May 14 Through November 15, 1964
Contract: NAS3-4174

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FOREWORD

This report describes the research activity carried out in fulfillment of Contract NAS3-4174 during the period from May 14, 1964 through November 15, 1964. The work was conducted under the direction of the Space Power Procurement Section, Lewis Research Center, National Aeronautics & Space Administration, with Robert L. Davies acting as Project Manager.

ABSTRACT

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The effect of a simulated space environment on selected high-emittance coatings is being evaluated. The coatings were applied to either AISI-310 stainless steel or columbium - 1 percent zirconium alloy. The results obtained to date indicate that calcium titanate and iron titanate are suitable materials for space radiator coatings.

During the first six months of the program, four total hemispherical emittance rigs used in a previous program were modified to permit specimens to be tested at temperatures up to 1800°F at pressures lower than 10^{-7} mm Hg for periods up to 5000 hours.

The selection of materials for long-term emittance testing was based on results from previous emittance programs conducted at Pratt & Whitney Aircraft and on the results of a study of the literature. Candidate materials were subjected to short-term emittance testing to confirm that their emittance was 0.85 or better and that their stability during exposure to high temperature and vacuum warranted extended testing. During the report period, coatings of calcium titanate, iron titanate, and a zirconium diboride-molybdenum disilicide composition were subjected to short-term emittance testing. The results indicated that calcium titanate and iron titanate were suitable for long term emittance testing.

Three specimens are being tested in the long-term total hemispherical emittance rigs. The first of these is AISI-310 stainless steel coated with calcium titanate. This test is being conducted at 1350°F and about 2000 hours have been accumulated to date. The emittance has been stable at 0.91 throughout the period. The second specimen is AISI-310 stainless steel coated with iron-titanate. A total of about 1000 hours with a steady emittance of 0.89 have been accumulated. The third specimen is columbium - 1 percent zirconium coated with iron titanate. The emittance for this specimen varied between 0.88 and 0.85 during the 1950 hours of testing at 1700°F accumulated to date. These tests will be continued until each specimen has been tested for 5000 hours.

Adherence tests were conducted on AISI-310 stainless steel specimens coated with 4-mil layers of calcium titanate. The adherence was evaluated by vibrating the specimen at a frequency of 120 cps for up to 10^{7} cycles with a maximum stress of 55,000 psi. There was no indication of coating separation or spalling during the test. The tests were terminated by failure of the substrate.

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I. INTRODUCTION

A program is being conducted to determine the suitability of selected high-emittance materials for use as coatings on nuclear space power-plant radiators. The coating materials are being evaluated at elevated temperatures and high vacuum for their emittance stability, adherence, and compatibility when applied to AISI-310 stainless steel or columbium - 1 percent zirconium. This report describes the work conducted during the first six months of the program.

The program is divided into five phases. Phase I consists of the modification of four total hemispherical emittance rigs used previously for testing coatings of SNAP-8 and Sunflower I test radiator sections at 700°F. When modified, the rigs are suitable for testing tubular specimens at temperatures up to 1800°F in vacuums of 10^{-7} mm Hg or better for periods of at least 5000 hours. For Phase II, selected materials coated on AISI-310 stainless steel or columbium - 1 percent zirconium tubes are tested for 250 hours at elevated temperatures to determine their suitability for long-term testing. During the 250-hour test, the specimens are thermally cycled after initial heating, and after 100 and 200 hours of testing to determine the adherence of the coating. The specimens are required to demonstrate an emittance of 0.85 or better before being accepted for long-term testing. Phase III is the 5000-hour emittance testing. Specimens with AISI-310 stainless steel substrates are tested at 1350°F and those with columbium - 1 percent zirconium substrates are tested at 1600 to 1800°F. Testing includes thermal cycling after 300, 400, and 500 hours. Phase IV is a room-temperature fatigue test program for the candidate coatings on AISI-310 stainless steel or columbium - 1 percent zirconium. For these tests, the specimens are vibrated at 120 cps for up to 10^7 cycles with various stress levels. Phase V consists of the testing of new, promising coatings recommended by the Contractor and approved by the NASA project manager. During all phases of the program, chemical and metallurgical examinations, including X-ray analysis, microhardness testing, and metallographic examinations, are conducted when warranted.

II. COATING MATERIAL SELECTION

A. Introduction

The initial selection of coating materials for long-term endurance testing was based on the results of previous emittance testing and on a study of new materials in the literature. Final selection was based on the results of short-term endurance tests and an evaluation of the chemical, metallurgical, and mechanical properties of the candidate materials.

During the previous program conducted under NASA contract NASw-104, over 70 materials were tested to evaluate their high-temperature emittance and adherence properties in vacuums of 10^{-6} mm Hg or higher. The materials tested included metals, metals with oxidized surfaces, and various refractory ceramic coatings. The coatings were applied by various techniques including thermal spraying, electroplating, and with liquid binders. The results of the previous program indicated that calcium titanate, iron titanate, nickel chrome spinel, and silicon carbide were the most promising materials of those tested for space radiator applications. These materials exhibited emittance values of 0.85 or better when tested in a vacuum of 10^{-6} or higher at elevated temperatures.

The search for new materials indicated that zirconium titanate, columbium titanate, and a zirconium diboride-molybdenum disilicide composition were promising.

B. Calcium Titanate

Ten calcium titanate coatings were tested in the total hemispherical emittance rigs during the previous program. The coatings were 1 to 5 mils thick and were applied by either plasma spraying or aluminum-phosphate bonding to AISI-310 stainless steel, columbium, or columbium - 1 percent zirconium. Testing was conducted at temperatures ranging from 300 to 2200°F. Emittance values ranging from 0.61 to 0.91 were measured with the lower values occurring either at very high temperatures or as a result of inadequate bonding. The tests indicated that calcium titanate coatings are quite stable in vacuums of 10^{-7} mm Hg or higher at temperatures up to 1450°F. The data also indicated that plasma spraying produced better calcium-titanate coatings than aluminum phosphate bonding.

C. Iron Titanate

Seven iron-titanate coatings plasma sprayed on columbium-1 percent zirconium tubes were tested during the previous program. These coatings were between 2 and 5 mils thick and were tested over the temperature range of 300°F to 2200°F. The data indicates that iron titanate is stable up to 1800°F for the short periods for which it was tested and that the emittance is generally in the range of 0.85 to 0.88.

D. Nickel-Chrome Spinel

Nickel-chrome spinel coatings were prepared for the previous program by heating stoichiometric mixtures of nickel oxide (NiO) and chromic oxide (Cr_2O_3) at 2500°F for 10 hours. One mix was fired in air and the resulting material was 70 percent spinel. A second mix was fired in an oxygen-rich atmosphere and the resulting mixture was 80 percent spinel. A total of five coatings were applied to columbium-1 percent zirconium by plasma spraying and tested. The results indicated that the coatings containing the higher spinel concentrations exhibited higher emittance values and that the material was stable at 1600°F for at least 260 hours. At 1800°F, however, the emittance dropped appreciably during a 100 hour test.

E. Silicon Carbide

Seven total hemispherical emittance tests were made during the previous program with silicon-carbide coatings aluminum-phosphate bonded to columbium-1 percent zirconium tubes. The coatings were prepared from 400 and 600 mesh silicon carbide powder and were 4 to 8 mils thick. Testing conducted over the temperature range of 300°F to 1700°F indicated that an emittance of 0.90 or better could be achieved, but that it was difficult to achieve adequate bonding. There was a substantial amount of scatter in the data. Silicon carbide bonded with aluminum phosphate was limited to temperatures below 1400°F, and in some cases, the binder lowered the emittance. Thermal spraying was not possible since silicon carbide has no melting or softening point. Consequently, silicon carbide was considered for the present program provided a suitable bonding technique could be developed.

F. New Materials

During this report period, a continuous literature search for new coating materials was conducted. In addition, several manufacturers and research institutes engaged in the development, manufacturing, or testing of refractory coatings were visited.

Several materials were found which appeared to have potential for space radiator coatings. Those chosen for additional study were zirconium titanate, columbium titanate, and a zirconium diboride-molybdenum disilicide composition. This last material was developed by the Carborundum Corporation, Niagara Falls, New York under the trade name "Boride Z." The emittance values of sintered, and hot-pressed specimens have been reported.[1]

G. Conclusions

On the basis of previous test results, a literature search, and a survey of the industry, calcium titanate, iron titanate, nickel chrome spinel, silicon carbide, zirconium titanate, columbium titanate, and a zirconium diboride-molybdenum disilicide composition were selected for additional study.

[1] I. M. Logan and J. E. Niesse, "Process and Design on a Boride-Silicide Composition Resistant to Oxidation to 2000°C," USAF Contract AF33 (616)-8014, ASD-TDR-62-1055, November 1962.

III. APPARATUS

A. Introduction

The basic apparatus used in this program were developed during previous contracts for the measurement of emittance of typical space radiator coatings. The equipment was designed to simulate a space environment for extended periods and to provide accurate emittance data. To fulfill these objectives, the test rigs were designed in accordance with the following requirements.

1. The test specimen must radiate to an adequately low-temperature, high-absorptance heat sink.

2. The energy dissipated by the test specimen must be accurately accountable.

3. The test chamber must operate at pressures no higher than 10^{-6} mm Hg in order that space conditions are essentially duplicated.

4. The system must be capable of operating continuously for periods greater than one year.

5. Sufficient instrumentation must be used to permit all specimen operating parameters to be determined accurately, and the instrumentation must be designed to ensure long-term reliability.

6. The system must be flexible so that a change in the specimen design or in test conditions can be easily accommodated.

Five of the rigs designed and built in accordance with these requirements for the previous program are available for use in the current program. These rigs have been modified so that each is capable of providing total hemispherical emittance data as a function of time and temperature. Tests of at least 250 hours duration are conducted in one of the rigs to screen coatings being considered for extended testing. The remaining rigs are being used for 5000-hour tests.

B. Short-Term Endurance Rig

The short-term endurance rig is used to determine emittance stability at selected temperatures for periods up to 300 hours. The rig includes a vacuum chamber, an evacuation system, an instrumentation flange, a specimen-heating power supply, and temperature measurement instrumentation. The rig is shown in Figure 1 and a schematic diagram is shown in Figure 2.

The vacuum chamber, which was made of AISI-304 stainless steel, is 13 inches long and has a 3-inch inside diameter. The inside wall was coated by brush with a "3M Black Velvet" coating to provide a low-reflectance radiation sink. This enamel coating was tested prior to this program and was found to have a total hemispherical emittance between 0.90 and 0.95 through the 200 to 700°F temperature range. The actual composition of the coating is held proprietary by the Minnesota Mining and Manufacturing Company.

A viewing port for specimen observation and optical measurements is located at the midpoint of the chamber. The port consists of an optically plane quartz window cemented to a stainless steel cell with epoxy resin. The cell assembly is bolted and sealed with a gold O-ring to a mating flange which is welded to a tubular extension from the chamber wall. The window is protected from condensate by a magnetically controlled rolling disk shutter installed in a slot between the cell and the flange. The chamber is wrapped with 3/16-inch diameter copper tubing for water cooling to minimize radiation from the walls. In addition, a heating element is wound over the cooling coil to bake out the chamber during initial evacuation. Flanges at both ends of the chamber provide for attachment of the vacuum manifold and for installation of the instrumentation flange. Vacuum sealing is accomplished by means of 0.020-inch diameter fully annealed gold O-rings compressed between polished sealing surfaces.

The vacuum manifold connects a 40-liter-per-second ion-gettering pump, a titanium sublimation pump, an ionization gauge, a cold trap assembly, and a roughing valve to the chamber. The manifold is designed such that no valves or ducts are located between the chamber and the pumps so that the optimum pumping speed for the system is achieved.

A mechanical roughing pump with a liquid nitrogen cold trap is used during bakeout and is also used to establish the starting condition for the ion-gettering and sublimation pumps. The manifold, ion-gettering pump, and the sublimation pump are all equipped with bakeout heaters to facilitate outgassing. Pressure during bakeout is monitored with a thermocouple vacuum gauge. During testing, pressures in the 10^{-8} mm Hg range are maintained by using the ion-gettering pump and the titanium sublimation pump. The walls of the sublimation pump are wrapped with 1/4-inch diameter copper tubing to provide water cooling and to increase the efficiency of the pump. The chamber pressure is determined by a Bayard-Alpert type ionization gage and by measuring the ion-gettering pump current and determining the pressure from calibration curves.

The instrumentation flange provides power feed throughs, thermocouple feed throughs, and the specimen mounting and positioning assembly. The mounting assembly has a thermal expansion take-up device which also positions the specimen away from the centerline of the chamber to minimize the effect of reflections from the chamber walls back onto the specimen. The instrumentation flange is equipped with terminals for three platinum - platinum 10 percent rhodium thermocouples and a set of voltage leads.

Specimen temperature is measured by thermocouples at temperatures below 1300°F and by thermocouples and an optical pyrometer focused on an integral black-body hole in the specimen at temperatures above 1300°F.

The specimen power supply consists of a multi-tap step-down power transformer, the primary of which is controlled by a variable transformer. Power measurements are made by measuring the voltage drop across the specimen test section and across a current shunt by means of an AC-DC differential voltmeter. Thermocouple outputs for temperature measurement are measured with a slide-wire millivolt potentiometer used in conjunction with an ice bath reference junction.

The short term endurance rig was modified for the present series of tests by the addition of a new vacuum manifold, titanium sublimation pump, ionization gauge, cold trap assembly, and roughing valve. The modifications enable lower ultimate chamber pressures to be attained, especially with specimens that outgas severely. The changes also provide an alternate method of chamber pressure measurement and enable simplifications in operating procedures. The increased size of the modified rig required that the rig be moved from its former location on the long term emittance rig bench to a rolling table on which the associated rig equipment could be mounted.

C. Long-Term Endurance Rigs

The long term endurance rigs are used for emittance stability tests at selected temperatures for periods in excess of 5000 hours. The facility containing the rigs is shown in Figures 3 and 4. Four rigs, each consisting of a vacuum chamber, an instrumentation flange, evacuating equipment, a power supply for specimen heating, and instrumentation for measuring power and temperature are available. At present, three of the rigs are in operation.

The vacuum chambers, shown in Figure 3, are 20 inches high, have inside diameters of 15 1/2 inches, and were fabricated from AISI-304 stainless steel. Permanent bake-out heaters and cooling coils are incorporated in the chamber walls. A 5.4-inch diameter window is provided for observation of the specimen and for obtaining radiation measurements. Magnetically controlled polished stainless steel shutters are used to prevent heating of the window by specimen radiation, to prevent reflection and radiation to the specimen, and to protect the window from condensate. A 3-inch square port is located 180° from the large window and is fitted with a Bayard-Alpert type ionization gauge for measuring chamber pressure.

The inside of each chamber is coated with a high absorptance coating. One chamber interior is coated with acetylene black in xylol, another is coated with nickel-chrome spinel plus silicon dioxide bonded with aluminum phosphate, and another is coated with silicon carbide plus silicon dioxide bonded with aluminum phosphate solution. These chambers were coated during a previous contract and the coatings were still serviceable. The fourth chamber had to be recoated and was sprayed with "3M Black Velvet" coating.

The instrumentation flanges are designed to be independent from the basic vacuum chambers. They contain the specimen supports, thermocouple and power feedthroughs, thermocouple standoffs, and a thermal expansion take-up device. The portion of the instrumentation flange external to the vacuum chamber contains the coolant manifold and the power and thermocouple connections. The instrumentation flange is shown in Figure 5.

The instrumentation flange has provisions for seven platinum-platinum 10 percent rhodium thermocouples and five chromel-alumel thermocouples. Three flanges were assembled using high-temperature lead solder and one flange was assembled using low-temperature silver solder.

A mechanical vacuum pump in conjunction with a liquid nitrogen cold trap is used during bake-out and pump-down of the system to the starting pressure of the 40-liter/second ion-gettering pumps. The ion-gettering pumps are bolted directly to the baseplates of the rigs and are used continuously during testing. The pumps are capable of producing pressures below 1×10^{-9} mm Hg and maintain pressures in the 10^{-8} mm Hg range with the specimens heated to endurance conditions. Gold O-rings, copper gaskets, and Teflon seals are used for sealing the various vacuum chamber components. The chamber pressure is determined both by a Bayard-Alpert type ionization gauge and by measuring the ion-gettering pump current.

All instrumentation and rig controls are contained in a control console as shown in Figure 4. The console is set up to provide centralized control for each rig as indicated in the block diagram in Figure 6. The control system for each rig includes a safety device which will automatically shut down the test in the event of a power failure or loss of cooling water. The test must be manually started after the power or water has been restored. The instrumentation system includes a multipoint millivolt recorder, which, by means of a time-controlled stepping relay, automatically records the chamber and specimen thermocouple outputs hourly. A manually operated slide-wire potentiometer is used to provide highly accurate temperature data for emittance calculations. Specimen voltages and currents are measured by a highly accurate AC-DC differential voltmeter.

The original rigs were designed to permit easy conversion for other types of testing. To adapt the rigs for the present test program, the only requirements were the replacement of the instrumentation flange and the addition of a step-down power transformer. In addition, an ionization gauge was added to the chamber to provide a supplementary method of chamber pressure measurement. The elements in the ion-gettering pumps were replaced to ensure reliability. The interior wall of one chamber was recoated. The instrumentation and control console required no modifications.

IV. SPECIMEN PREPARATION

A. Description

The specimens used for the total hemispherical emittance and endurance testing consist of coated tubes which are 9 inches long, 0.250 inch in diameter, and have a wall thickness of 0.010 inch. The tubes used for this study were made of either AISI-310 stainless steel or columbium-1 percent zirconium alloy. These alloys were ordered to the specifications shown below.

Columbium - 1 Percent Zirconium	
Columbium	98.5% minimum
Zirconium	0.8 to 1.2%
Carbon	100 ppm maximum
Nitrogen	300 ppm maximum
Oxygen	300 ppm maximum
Hydrogen	20 ppm maximum

AISI-310 Stainless Steel	
Chrome	24.00 to 26.00%
Nickel	19.00 to 22.00%
Manganese	2.00% maximum
Silicon	1.50% maximum
Carbon	0.25% maximum

The emittance tubes have two black-body holes 0.0235 inch in diameter through one wall at the midpoint. The holes are separated longitudinally by 0.0522 inch and displaced laterally by 0.043 inch from the tube centerline. The tubes are thoroughly cleaned in a saturated solution of concentrated sulfuric acid and potassium dichromate to remove organic contaminates before the coating is applied. The tubes are then grit blasted with 60 mesh silicon carbide at 100 psi. to a roughness height of 110 microinches as determined by a profilometer using arithmetic averaging.

B. Plasma-Arc Spraying

All of the coatings for this program are applied by plasma-arc spraying. Plasma-arc spraying utilizes the high energy of an electric arc to ionize a gas and form a plasma. The energy of the plasma is released as heat. The coating material, in powder form, is propelled through the plasma, where it absorbs heat and melts, toward the specimen surface. The high temperatures attainable and the non-oxidizing atmosphere provided by plasma spraying are more suitable for the intended applications than the conditions produced by flame spraying. The coatings tested were applied by Pratt & Whitney Aircraft using Plasmadyne equipment.

After being coated, the tubular specimens are flattened for 3/4 inch at each end to facilitate clamping to the power electrodes. The flattening process results in the removal of most of the coating in the clamping region and any remaining residue is removed by means of an abrasive to ensure good electrical contact. A few flakes of the coating removed in this operation are saved and analyzed for comparison with the analysis of the coating after the test.

The black-body holes are reamed with a drill to remove any coating material that might have accumulated during the coating process.

C. Specimen Instrmentation

The location for thermocouple and voltage lead attachment points are marked on each specimen. The coating is then removed from a rectangular area, 0.010 x 0.030 inch, at each location by means of a tungsten-carbide scraping tool. The thermocouple and voltage leads are attached by resistance welding to the substrate metal with a capacitive discharge welder. The distance between the voltage leads is measured with a measuring microscope to allow precise calculation of the test section radiating area.

The specimens for short-term endurance tests are normally instrumented with three platinum-platinum 10 percent rhodium thermocouples and two voltage leads. The specimens for long-term endurance tests are instrumented with seven platinum-platinum 10 percent rhodium thermocouples and five chromel-alumel thermocouples. For these specimens, the thermocouple wires are used as voltage leads.

D. Specimen Installation

The instrumented specimen is clamped into the flange between the specimen end clamps and held under tension by the thermal expansion take-up device. The thermocouple wires are attached to the thermocouple standoffs by resistance welding. The chamber is then vacuum sealed and pumped down with a mechanical roughing pump backed by a liquid-nitrogen cold trap. The bakeout heaters are turned on and the chamber is baked out at 350°F for a period ranging from two to twenty-four hours depending on the degree of contamination. During bakeout, the chamber pressure is monitored with a thermocouple vacuum gauge. After bakeout is completed, the ion-gettering pump is started and the valve to the roughing pump and cold trap is closed. The system is allowed to cool, and, generally, ultimate pressures in the 10^{-9} mm Hg range are obtained within 16 hours after starting the ion-gettering pump. The specimen is then heated to the desired operating temperature. During testing, the chamber walls are water cooled to maintain a constant sink temperature.

The power input to the specimen is determined by measuring the voltage drop across a calibrated shunt to obtain the current and by measuring the voltage drop across the test section. Both measurements are made using an AC-DC differential voltmeter. The length and location of the test section are determined by the position of the voltage leads. The instrumentation circuitry of the long-term endurance rigs allows the use of any two thermocouple wires for voltage leads.

The specimen and chamber wall temperatures are determined by measuring thermocouple outputs with a slide-wire millivolt potentiometer used in conjunction with an ice bath reference junction. At temperatures above 1300°F, the specimen temperature is also measured using an optical pyrometer focused on one of the integral black-body holes in the specimen.

After the emittance testing is completed, the rigs are vented to atmospheric pressures using dry nitrogen. The specimens are removed from the rigs and subjected to additional testing and evaluation after which they are stored in transparent plastic tubes for future reference.

V. EMITTANCE CALCULATIONS AND ERROR ANALYSIS

The total hemispherical emittance is determined from the data by using the following equation:

$$\epsilon_{th} = \frac{IV}{\sigma A(T_s^4 - T_o^4)}$$

where:

ϵ_{th} = total hemispherical emittance

I = current through the test section

V = voltage drop across the test section

σ = Stefan - Boltzmann constant

A = surface area of the test section radiating power IV

T_s = temperature of the test section

T_o = temperature of the chamber wall

The maximum instrumentation error for each of the measured quantities is estimated as follows:

	Estimated Error (Percent)
Specimen heating current	±0.2
Test section voltage drop	±0.2
Test section radiating area	±0.25
Thermocouple measurement of test section temperature	±0.5
Optical pyrometer measurement of test section temperature	±0.4

Since errors from other causes are negligible, the maximum errors in reported total hemispherical emittance values obtained with the present rigs are ±2.7 percent when temperatures are measured by thermocouples and ±2.3 percent when temperatures are measured by an optical pyrometer.

VI. SHORT-TERM ENDURANCE EMITTANCE TESTS

A. Introduction

Those materials which appeared to be most promising on the basis of previous testing and the preliminary screening for chemical, mechanical, and metallurgical properties discussed in section VII of this report were subjected to short-term emittance testing as a final evaluation of their suitability for long-term testing. The materials tested were iron titanate, calcium titanate, and the zirconium diboride-molybdenum disilicide composition.

All short-term testing was performed in the short-term total hemispherical emittance rig. During the tests, the specimens were subjected to thermal shocks produced by shutting off the power to the specimen and allowing it to cool to the sink temperature. The specimen cooling rate is shown in Figures 7 and 8 for starting temperatures of 1350°F and 1700°F respectively. Subsequently, the specimen was slowly returned to the operating temperature.

B. Test Results

Iron Titanate - Two short-term endurance tests were performed on columbium-1 percent zirconium tubes coated with a 4-mil layer of iron titanate.

The first test was conducted at 1800°F in a vacuum of about 10^{-8} mm Hg. The test was run for 29.8 hours when it was terminated because of a separation of a voltage lead from the specimen. During the test, the total hemispherical emittance was 0.86.

The second test was conducted for 200 hours at 1700°F and 118 hours at 1800°F. The emittance values obtained during this test ranged from 0.86 to 0.88. The emittance remained steady at 0.88 for the last 100 hours. Visual examination of the specimen after testing revealed no changes in the specimen as a result of testing. The coating texture was the same as it was before testing, and no indications of cracking or spalling were present. Examination of the rig showed that no coating volatilization occurred. This specimen, therefore, was selected for testing at 1700°F for 5000 hours in one of the long-term hemispherical emittance endurance rigs.

An AISI-310 stainless steel tube coated with a 4-mil layer of iron titanate was endurance tested at 1350°F for 263 hours in a vacuum of about 10^{-8} mm Hg in the short-term hemispherical emittance rig. The emittance values obtained were about 0.90 but were somewhat erratic. This erratic behavior was later found to be a result of faulty instrumentation. In order to be certain of the emittance, an additional 44-hour test was conducted at 1350°F after reinstrumentation. A constant emittance value of 0.89 was measured. This specimen was subjected to thermal cycling after initial heating, after 100 hours, and after 200 hours.

The tested specimen was thoroughly examined after the short-term hemispherical endurance test. No change in the appearance of the coating and no cracking or spalling of the coating from the substrate were apparent. This specimen was, therefore, selected as the second specimen for a 5000-hour test in the long-term hemispherical endurance rig. The operating temperature was to be 1350°F.

The iron titanate powder used for these tests was obtained from the Continental Coating Company. The particle size distribution of the powder as received is shown in Table 1. Analysis made of the powder before use showed no crystalline phase other than iron titanate (Fe_2TiO_5) to be present. Emission spectrographic analyses detected the presence of 0.5 percent Mn, 0.1 percent Mg, 0.7 percent Al, and 0.03 percent Cu.

TABLE 1

Particle Size of Iron Titanate Powder

Particle Size (Microns)	Cumulative Weight Percent
66	0
52	15.0
41	24.0
33	38.0
26	39.0
21	50.0
17	57.0
15	65.0
13	71.0
10	81.0
8	91.0
6	100

The coatings were applied by Pratt & Whitney Aircraft by plasma spraying using Plasmadyne equipment. Argon was used for both the arc gas and powder carrier gas with respective flow-rates of 45 and 40 cubic feet per hour. The current used was 450 amperes. Before the coatings were applied, the substrate was heated to approximately 250°F by blasting the specimen with the argon carrier gas. The distance from the gun to the substrate was approximately three inches.

The iron titanate coating was analyzed after it was plasma sprayed onto the columbium-1 percent zirconium tubes. The material for analysis was obtained from the end of the columbium-1 percent zirconium tube. The X-ray diffraction analysis detected only iron titanate crystal phase. The emission spectrographic analysis detected the same elements in about the same percentages as previously.

Calcium Titanate - An AISI-310 stainless steel tube coated with a 4-mil layer of calcium titanate was tested for 265 hours at 1350°F. The total hemispherical emittance data obtained during the first part of the test was 0.88. The emittance increased to 0.89 after 200 hours and maintained this value for the final 65 hours. Thermal cycling tests were performed after initial heating, after 100 hours, and after 200 hours.

Visual examination of the specimen after removal from the short-term emittance rig showed no change on the coating texture, and there was no indication of cracking or spalling. Examination of the short-term rig revealed no volatilization of the coating during testing. This specimen, therefore, was selected as the third specimen for long-term endurance testing. The operating temperature was to be 1350°F.

A columbium-1 percent zirconium tube was coated with a 5-mil layer of calcium titanate and tested at 1350°F and 1650°F in the short-term endurance rig at a vacuum of 10^{-8} mm Hg. The emittance was 0.88 during the 100-hour 1350°F test. However, when the temperature was raised to 1650°F, the emittance decreased to 0.62 during a 68-hour test. The drop in emittance is attributed to a chemical breakdown of the coating.

The columbium-1 percent zirconium tube was analyzed and a substantial amount of oxygen was found to have diffused into the metal. The oxygen content found by vacuum fusion testing was about 3200 ppm in the columbium alloy. Unused columbium tubes usually have less than 200 ppm of oxygen. The results from this test indicate that calcium titanate is not useful in the 1600 to 1800°F temperature range and this material, therefore, has not been given further consideration for high-temperature testing.

The calcium titanate powder used for these tests was obtained from the Titanium Division of National Lead Corporation. The particle size distribution of the powder is shown in Table 2. The X-ray diffraction analysis performed showed $CaTiO_3$ to be the only crystalline phase present. The emission spectrographic analysis showed the only other elements present to be silicon and magnesium in trace amounts.

TABLE 2

Particle Size of Calcium Titanate Powder

Particle Size (Microns)	Cumulative Weight Percent
66	23.0
52	58.0
41	80.0
33	81.0
26	87.0
21	93.0
17	96.5
15	96.7
13	97.0
10	98.6
8	99.0
6	100

The calcium titanate powder was applied to the AISI-310 stainless steel tubes and columbium-1 percent zirconium tubes by plasma spraying. Argon was used as both the arc gas and powder carrier gas. The spray flow rates were 55 and 30 cubic feet per hour, respectively. The current was 500 amperes.

Some of the sprayed powder was scraped from the AISI-310 stainless steel tube for analysis. The only crystalline phase detected was $CaTiO_3$. The spectrographic analyses showed trace amounts of silicon and magnesium plus slight amounts of iron, chromium, and nickel. The presence of iron, chromium, and nickel is attributed to small amounts of the stainless steel substrate being present in the sample because of the scraping operation used in obtaining powder for analyses.

Zirconium Diboride-Molybdenum Disilicide - A columbium-1 percent zirconium tube with a 4-mil thick coating of 90 mole percent zirconium diboride (ZrB_2) with 10 mole percent molybdenum disilicide ($MoSi_2$) in solid solution was tested. The emittance of this coating was under 0.85, and problems were encountered with inadequate adherence between the coating and the substrate during thermal cycling. The "Boride Z" evaluated by Pratt & Whitney Aircraft was applied by plasma-spraying after it had been crushed and screened to the desired -250 + 325 mesh size.

VII. CHEMICAL, METALLURGICAL, AND MECHANICAL STUDIES

Analyses were conducted to further evaluate iron-titanate-coated and nickel-chrome-spinel-coated columbium-1 percent zirconium. In addition, attempts were made to satisfactorily bond silicon carbide to columbium-1 percent zirconium.

A columbium-1 percent zirconium tube coated with iron titanate and tested during the previous program at 1800°F for 200 hours was chemically analyzed. The results indicated that no significant changes had occurred in either the coating or the substrate. Oxygen diffusion analysis showed only a slight increase in the oxygen content of the substrate. Microhardness measurements obtained on a cross-section of the substrate tube revealed no significant hardness change. These results in conjunction with previous emittance tests and the recent short-term endurance test indicated the suitability of this coating-substrate combination for long-term endurance testing.

A similar analysis was conducted on a nickel-chrome-spinel coated columbium-1 percent zirconium tube. This tube, which was also evaluated during the previous program, was tested for 100 hours at 1800°F. It was found that about 2000 ppm of oxygen had diffused into the substrate and that the substrate hardness had changed significantly. These results indicated that nickel-chrome spinel coatings of the purity obtained to date were not suitable for high-temperature space radiator applications.

Attempts were made to apply silicon carbide to columbium-1 percent zirconium tubes by vapor deposition. This technique was not successful, however, and the coating spalled during the cooling cycle. The spalling was apparently caused by the differential thermal expansion between the coating and the substrate since the deposition process produced a dense, homogeneous coating. Additional testing of silicon carbide will not be conducted unless a method for satisfactorily bonding this material to a suitable substrate is developed.

VIII. LONG-TERM ENDURANCE EMITTANCE TESTS

A. Introduction

Three coating-substrate combinations tested in the short-term endurance emittance rig displayed adequate adherence and total hemispherical emittance values greater than 0.85 and consequently were selected for 5000-hour endurance testing. These combinations were iron titanate applied to columbium-1 percent zirconium, iron titanate applied to AISI-310 stainless steel, and calcium titanate applied to AISI-310 stainless steel.

B. Test Results

Iron Titanate Coating on Columbium-1 Percent Zirconium - The columbium-1 percent zirconium tube coated with a 4-mil layer of iron titanate has been tested for over 1950 hours at 1700°F in a vacuum of 10^{-7} mm Hg. or better. The emittance obtained to date for this coating is shown in Figure 9. As can be seen in this figure, the emittance of this coating dropped slightly from 0.88 to 0.85 between 600 and 1800 hours. This gradual decrease in emittance appears to have ceased and a constant value of 0.85 was observed at the end of this reporting period.

The vacuum which has been maintained during the run is shown in Figure 10. As can be seen in the graph, several slight leaks have occurred during the test. These were quickly stopped, and at no time has the pressure been higher than 10^{-7} mm Hg.

The specimen was thermally cycled after initial heating and after 100, 200, 300, 400 and 500 hours. Four additional unscheduled thermal cycles occurred between 1870 and 1950 hours. These were a result of the power being automatically shut off because of fluctuating water pressure. No adverse effects were noted either in emittance or appearance of the specimen after the thermal cycles. The appearance of the specimen after 1031.3 hours of testing is shown in Figure 11.

Iron Titanate on AISI-310 Stainless Steel - The AISI-310 stainless steel tube coated with a 4-mil iron titanate coating has accrued almost 1000 hours of endurance testing at 1350°F. The emittance values obtained to date are shown in Figure 12. As shown, the emittance has been steady at about 0.89 throughout most of the test.

The vacuum has been relatively constant at about 2 x 10^{-8} mm Hg. The vacuum maintained is shown in Figure 13. The appearance of the specimen shortly after the test began is shown in Figure 14.

The specimen was thermally cycled after 300, 400 and 500 hours to supplement the three cycles performed during the short-term test. No adverse effects were noted after the thermal shock tests.

Calcium Titanate Coating on AISI-310 Stainless Steel - The AISI-310 stainless steel tube with a 4-mil calcium titanate coating has been tested for about 2000 hours at 1350°F. The total hemispherical emittance has remained near or at 0.91 throughout most of the test as shown in Figure 15. The appearance of the specimen after 1077.4 hours is shown in Figure 16. This specimen was also thermally cycled after 300, 400, and 500 hours of testing. These three thermal cycles, along with the three thermal shock tests performed during the short-term test, had no noticeable effect on the emittance or adherance of the coating to the substrate.

The vacuum that has been maintained during the test is shown in Figure 17. The vacuum has been in the 10^{-8} mm Hg range for most of the test. At only one time did the pressure reach the 10^{-7} mm Hg. level. This was a consequence of a leak which developed in the thermocouple feed-through area of the instrumentation flange and was quickly corrected.

IX. ADHERENCE TESTS

A. Introduction

The ability of a high-emittance coating to dissipate heat from a space radiator is dependent upon the maintenance of an intimate contact between the coating and the substrate. Separation breaks the heat conduction path between the substrate and the coating and destroys the usefulness of the coating. Space radiators are subject to vibrations and these vibrations tend to break the coating-substrate bond. Consequently, an investigation is being conducted to determine the adherence of the high-emittance coatings being considered to AISI-310 stainless steel and to columbium-1 percent zirconium.

The adherence is evaluated by determining the number of cycles of 120 cps vibration required to fail a specimen at a given stress level. The specimen is considered to have failed when the coating either spalls, cracks, or separates from the substrate or when the specimen fractures. The maximum stress is calculated from the specimen geometry, the specimen modulus of elasticity, and the amplitude of vibration. The results are plotted as stress vs number of cycles to failure and the curve produced describes the adherence of the coating-substrate combination.

B. Fatigue Test Apparatus

Westinghouse high-temperature vibration fatigue equipment, Type MD, was used for all adherence testing. This apparatus, which is shown in Figure 18, is a fixed-cantilever, constant-deflection-type device designed to stress metal specimens in alternating bending at a frequency of 120 cycles per second. The specimen is driven at constant amplitude by a reciprocating electro-magnetic motor supplied with two-phase 60-cycle power. Controls are provided to maintain a constant amplitude of vibration of the specimen. An automatic control turns off the power when the specimen fractures.

The specimen is clamped at the top to a stationary frame and at the bottom to a drive rod connected to the reciprocating drive motor. Since the specimen is driven at 120 cps, small tuning weights are added to the lower clamp to bring the fundamental frequency of the cantilevered specimen and clamp assembly to about 120 cps. This reduces the required driving force. A microscope is used to measure the amplitude.

C. Specimens

The Westinghouse type fatigue specimens used were fabricated from AISI-310 stainless steel. Figure 19 shows the geometry of the specimen used in this program, and Figure 20 shows an actual 3.5-inch stainless-steel sample.

The specimens were prepared for coating by grit blasting the necked down section with number 60 silicon carbide grit at 100 psi, which is the same preparation procedure used for emittance specimens. Figure 21 shows a specimen after grit blasting. They were then coated by plasma-arc spraying using gas and current settings identical to those used in spraying emittance specimens. A coated fatigue specimen is shown in Figure 22.

During the report period, a total of ten samples were coated with calcium titanate and six were grit blasted and not coated for comparison.

D. Test Results

The six grit-blasted specimens which were not coated were used to determine the fatigue strength of AISI-310 stainless steel. Test results are depicted in Figure 23. The run-out fatigue strength (the stress which causes fracture in 10^7 cycles) was determined to be 50,000 to 55,000 psi.

To detect the initiation of coating failure, the test was stopped periodically and the specimen was examined. In all cases, the substrate failed before the coating showed any indication of failure. The run-out stress level (see Figure 24) was about 55,000 psi, the same as for the uncoated specimen. Figure 25 shows a specimen which was tested at a stress level above 55,000 psi and failed before 10^7 cycles. A photomicrograph of the coating and substrate is shown in Figure 26. The coating is seen to be completely intact and there is no evidence of cracking. Fatigue testing has shown that the adherence of the calcium titanate to the AISI-310 stainless steel is very satisfactory. The test results obtained indicate that this coating is capable of functioning for the limit of the substrate.

X. CONCLUSIONS

The work conducted to date indicates that calcium titanate and iron titanate are suitable materials for space radiator radiator applications.

AISI-310 stainless steel coated with calcium titanate was found to exhibit the following properties.

1. A total hemispherical emittance of about 0.91 for 2000 hours at 1350°F in a vacuum of about 10^{-8} mm Hg.

2. Freedom from adverse effects when subjected to thermal cycling.

3. Coating integrity when subjected to fatigue testing up to 10^7 cycles at 120 cps at stress loads up to 55,000 psi.

For AISI-310 stainless steel coated with iron titanate, the following properties were found.

1. A constant total hemispherical emittance of 0.89 for 1000 hours at 1350°F in a vacuum of 2 x 10^{-8} mm Hg.

2. Freedom from adverse effects when subjected to thermal cycling.

The properties demonstrated for columbium-1 percent zirconium coated with iron titanate were as follows.

1. A total hemispherical emittance between 0.85 and 0.87 for 1950 hours at 1700°F in a vacuum of about 10^{-8} mm Hg.

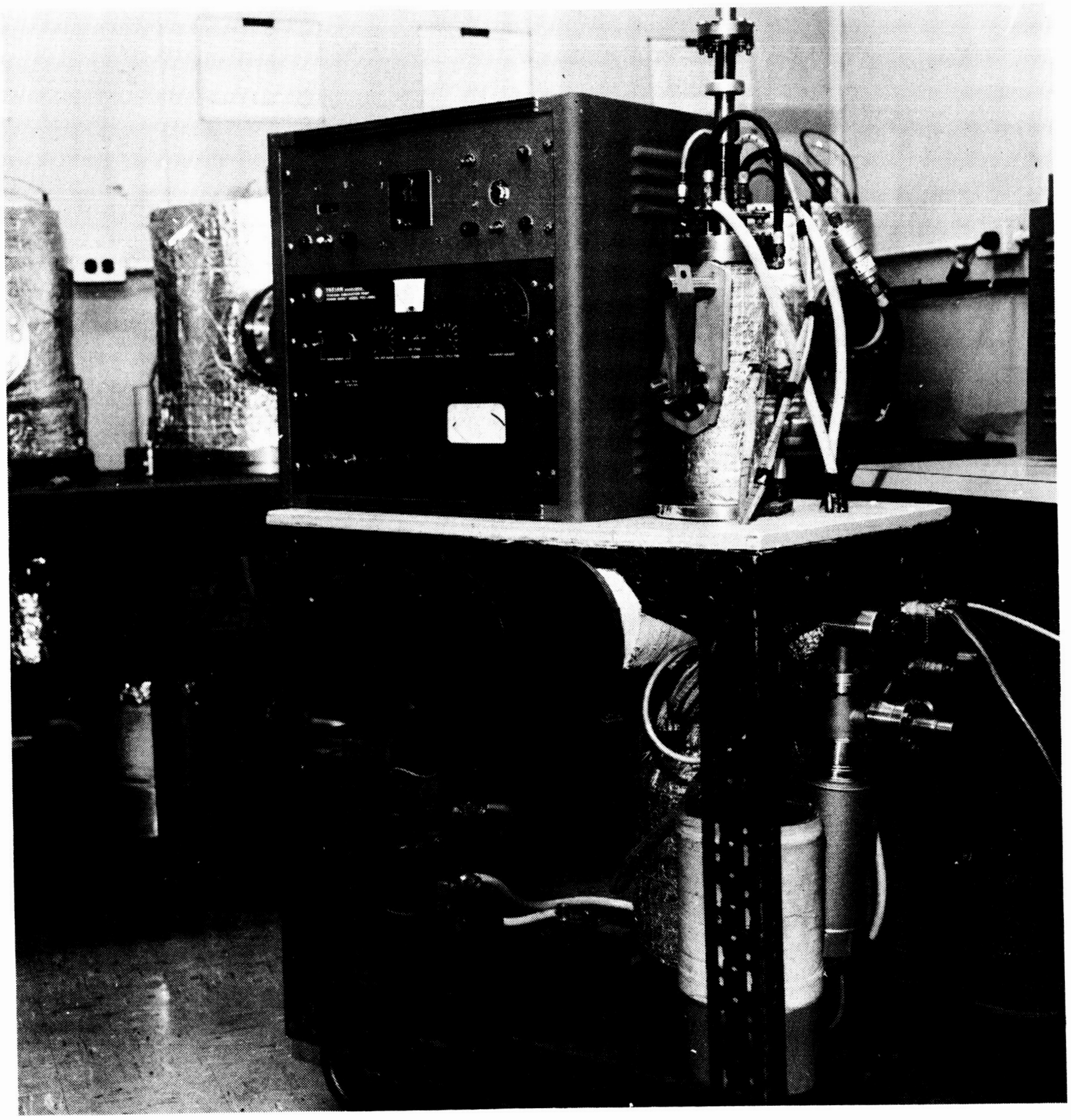
2. Freedom from adverse effects when subjected to thermal cycling.

XI. FUTURE WORK

The three 5000-hour total hemispherical emittance tests commenced during the report period will be continued.

Short-term endurance tests will be conducted on columbium titanate and zirconium titanate coatings applied to columbium-1 percent zirconium tubes. These tests will be conducted at 1700°F in vacuums of 10^{-7} mm Hg or higher. If one of these coatings exhibits high emittance and adequate adherence, it will be subjected to long-term endurance testing.

Adherence tests will be conducted on iron-titanate-coated AISI-310 stainless steel and columbium-1 percent zirconium specimens. Four-mil thick coatings will be used.



Short-Term Endurance Total Hemispherical Emittance Rig

Figure 1

SKETCH OF THE SHORT TERM ENDURANCE RIG SHOWING THE RELATIVE LOCATION OF SPECIMEN AND RIG DETAIL

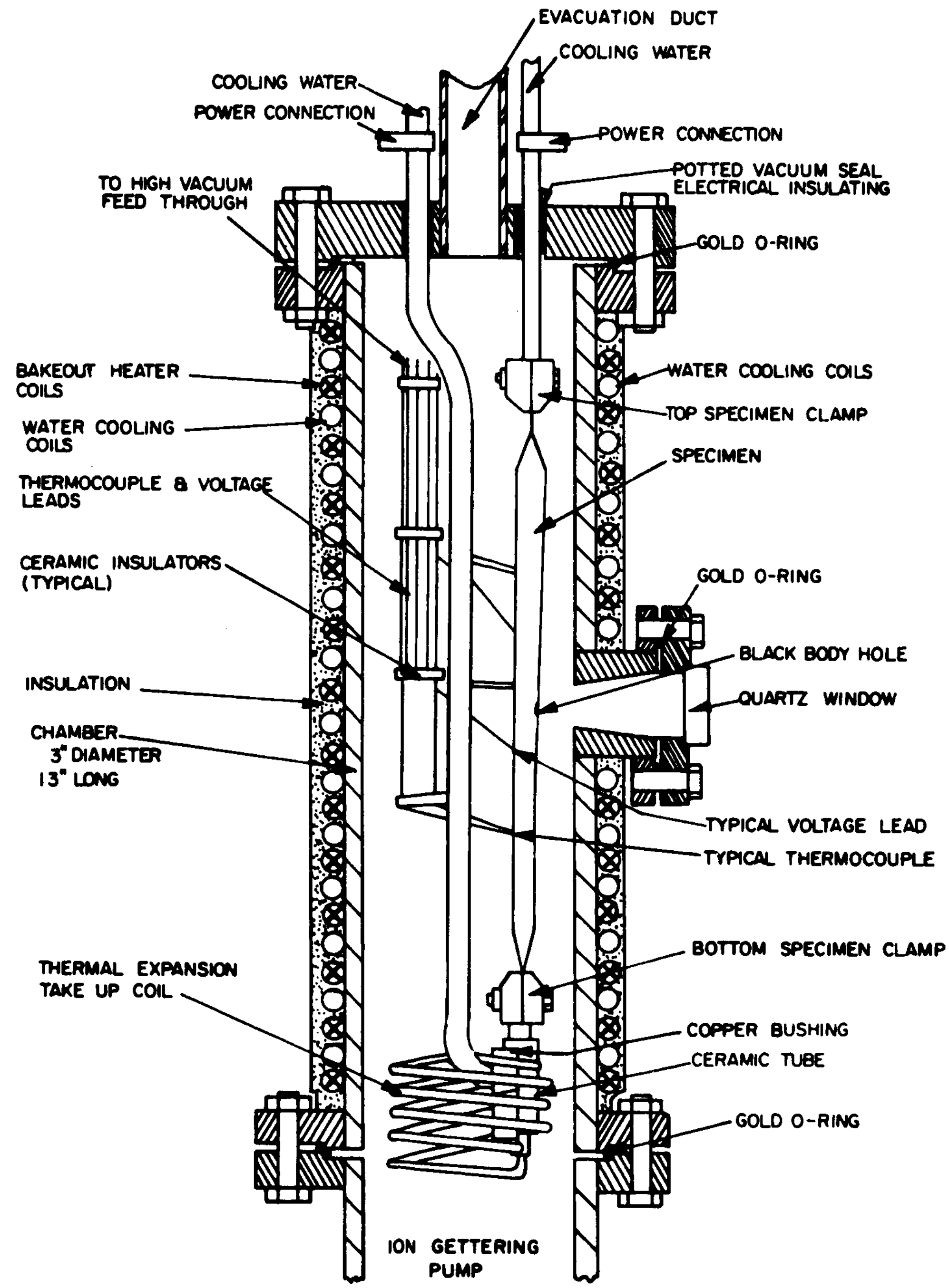
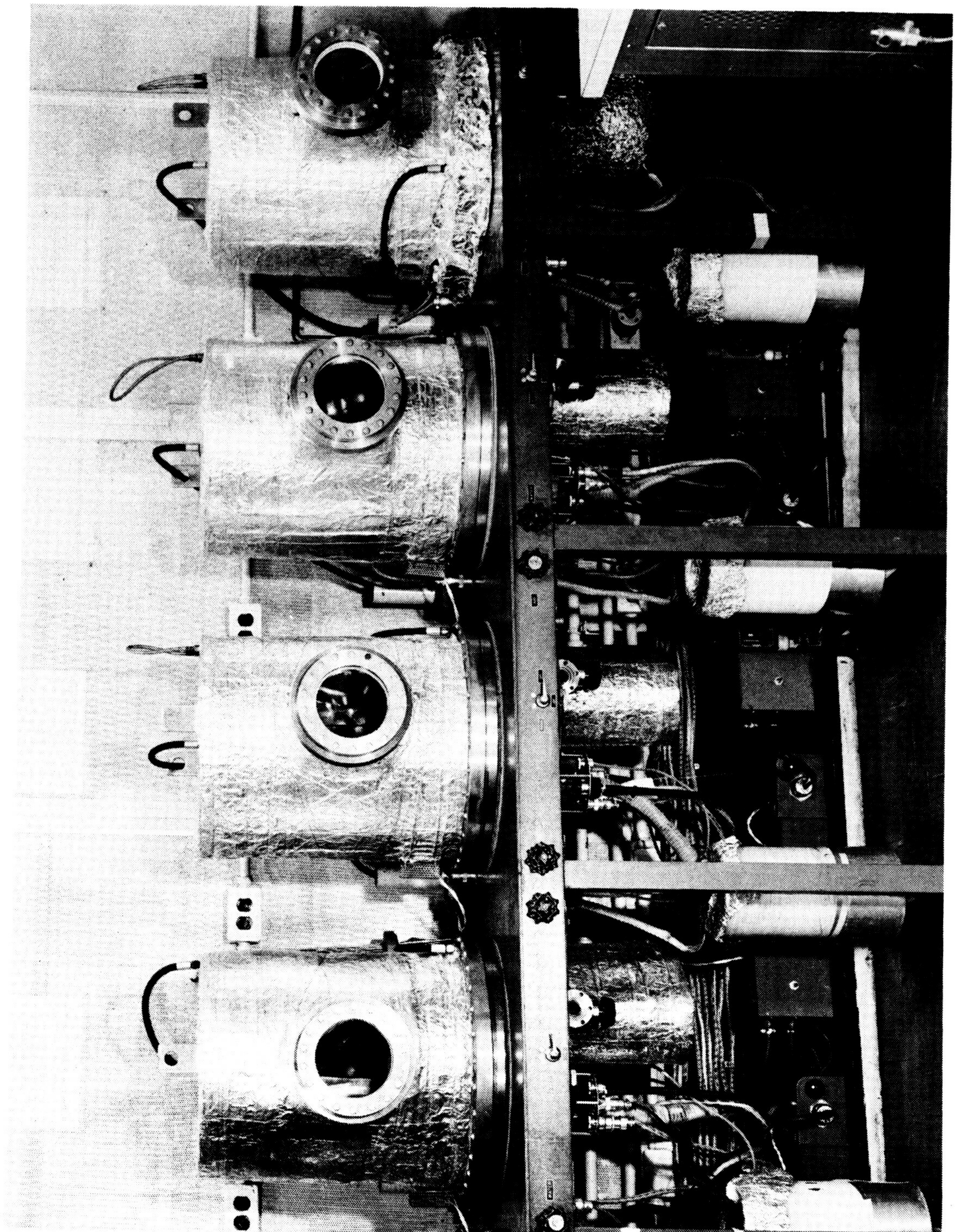
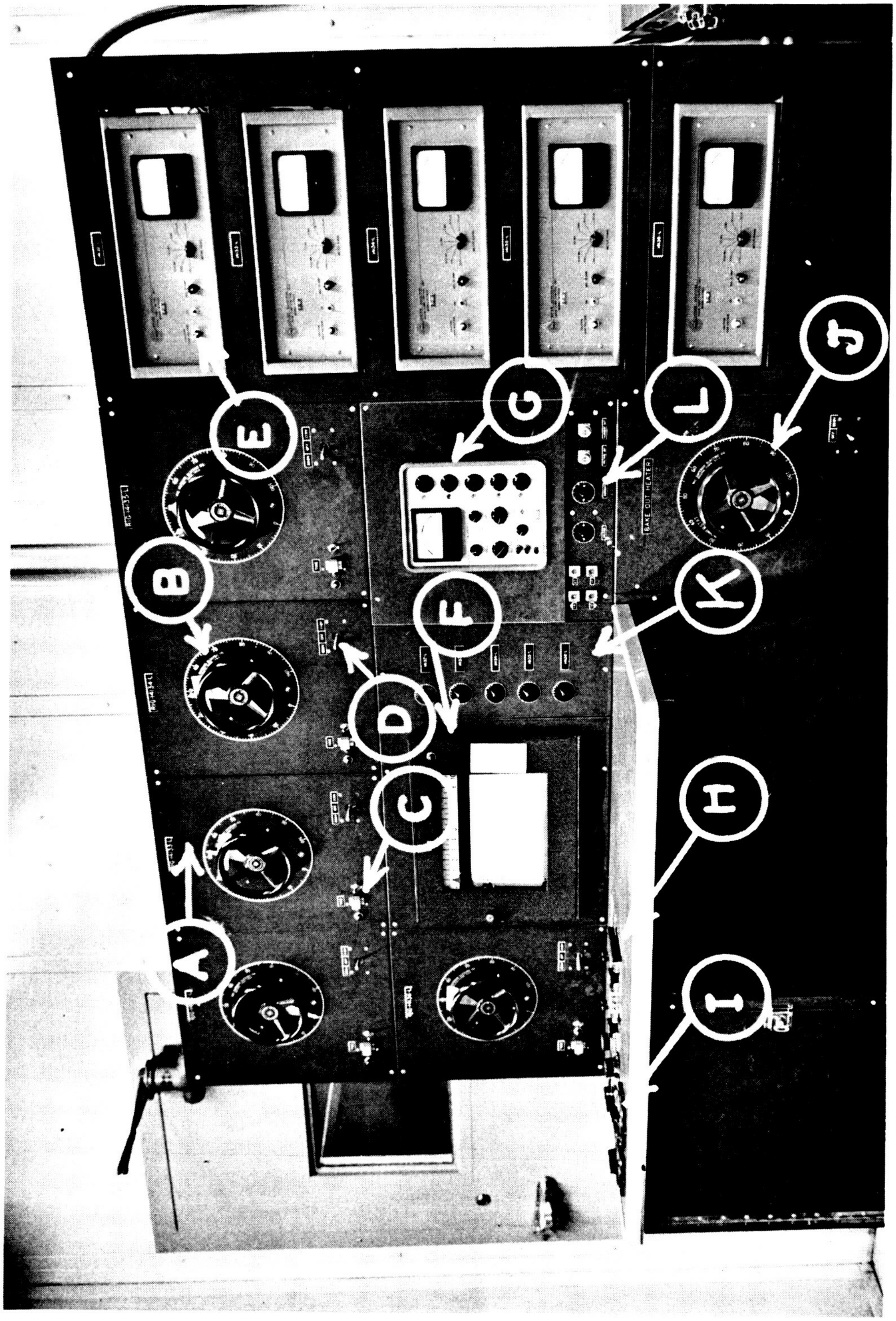


Figure 2



Long-Term Endurance Total Hemispherical Emittance Rigs

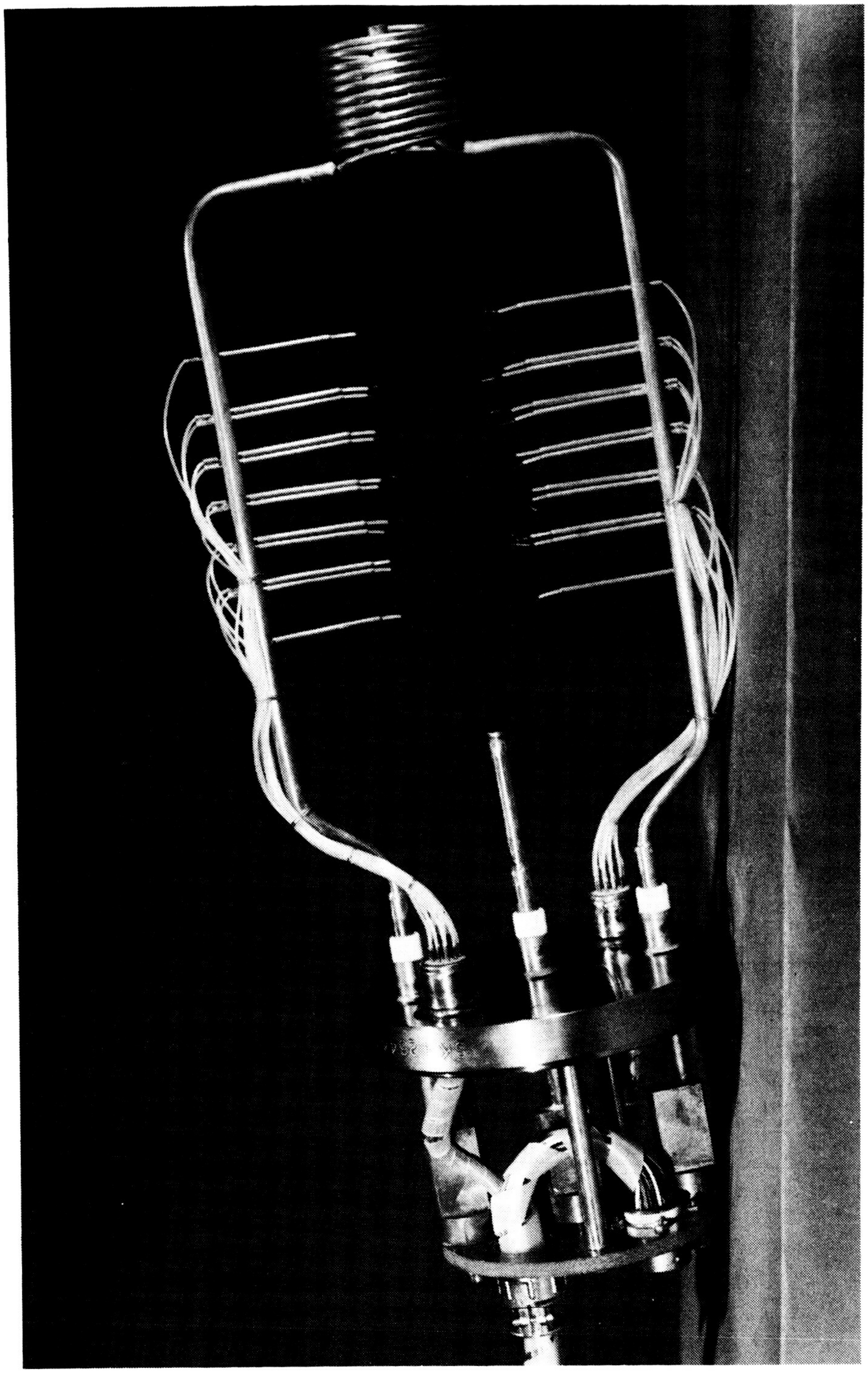
Figure 3



Control and Instrumentation Console for Endurance Rigs

A - Rig Control Panel
B - Rig Voltage Control
C - Rig Timer
D - Voltage Switch
E - Vacuum Pump Power Supply
F - Multipoint Recorder
G - AC-DC Differential Voltmeter
H - Potentiometer
I - Thermocouple Selector Switch
J - Bake Out Heater Control
K - Thermocouple Mode Switch Panel
L - Specimen I and V Selection Panel

Figure 4



Instrumentation Flange for Long-Term Endurance Rig

Figure 5

BLOCK DIAGRAM OF INSTRUMENTATION & ACCESSORY EQUIPMENT FOR ENDURANCE RIGS

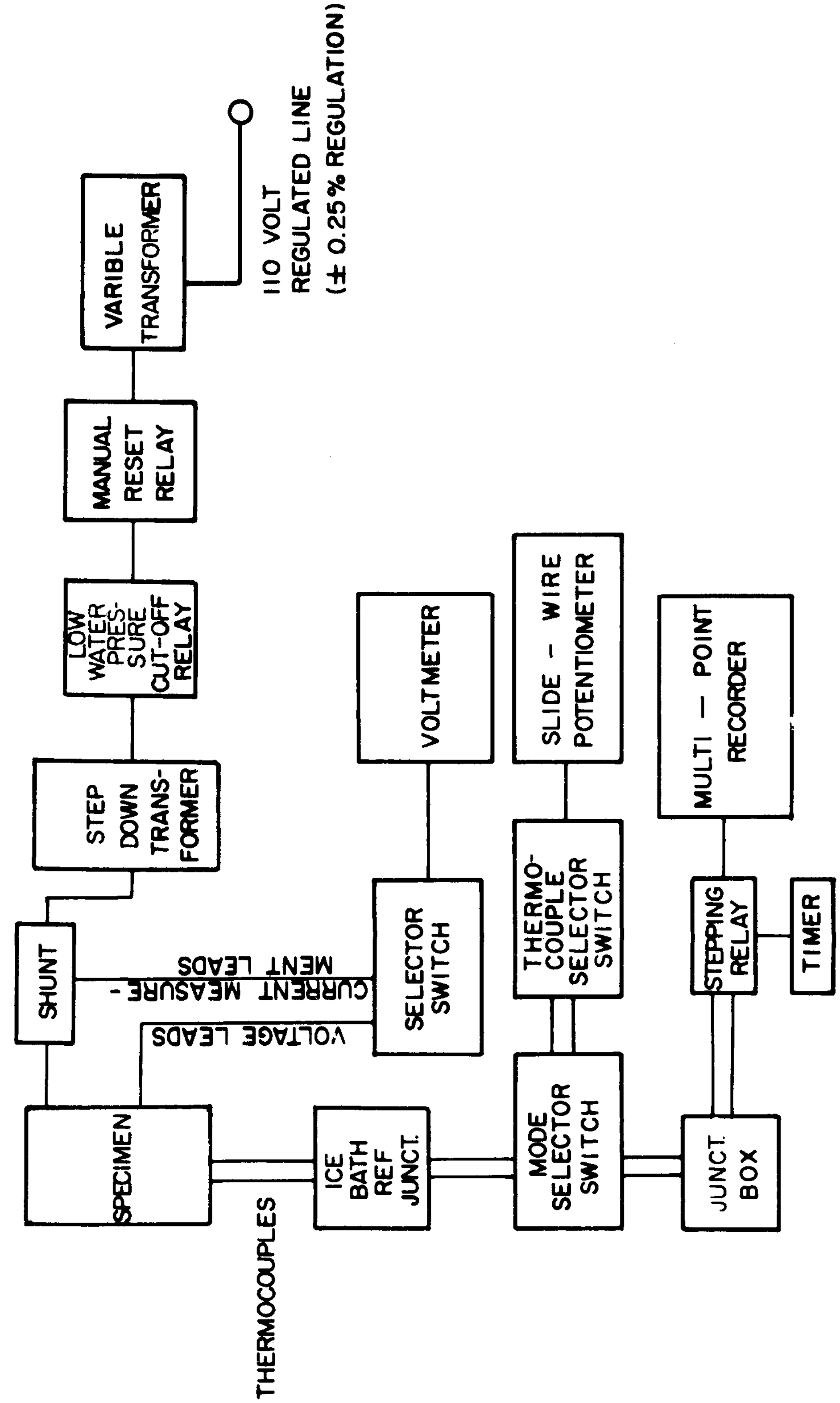


Figure 6

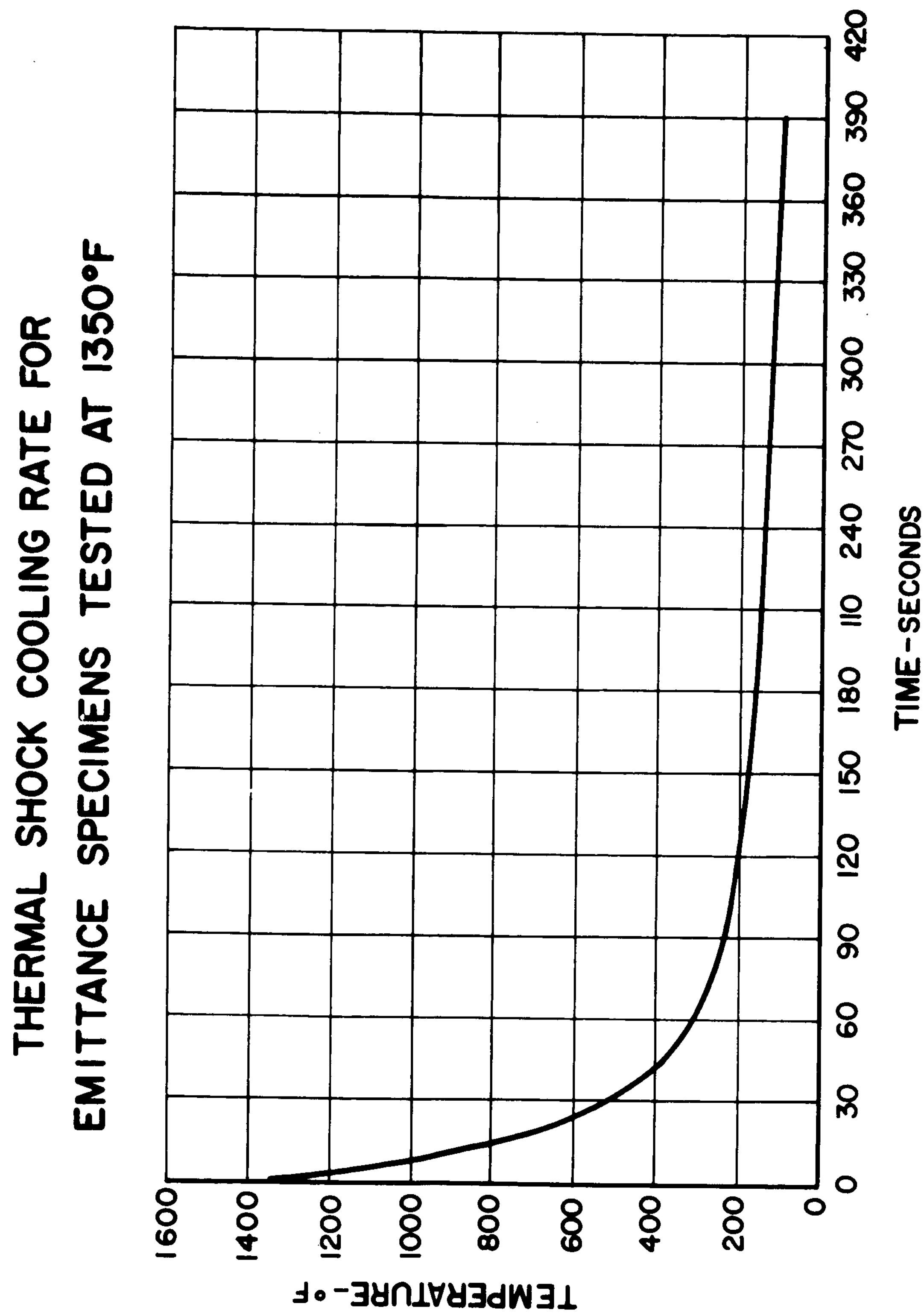

THERMAL SHOCK COOLING RATE FOR
EMITTANCE SPECIMENS TESTED AT 1350°F
TIME-SECONDS
0
30
60
90
120
150
180
110
240
270
300
330
360
390
420
TEMPERATURE-°F
0
200
400
600
800
1000
1200
1400
1600

Figure 7

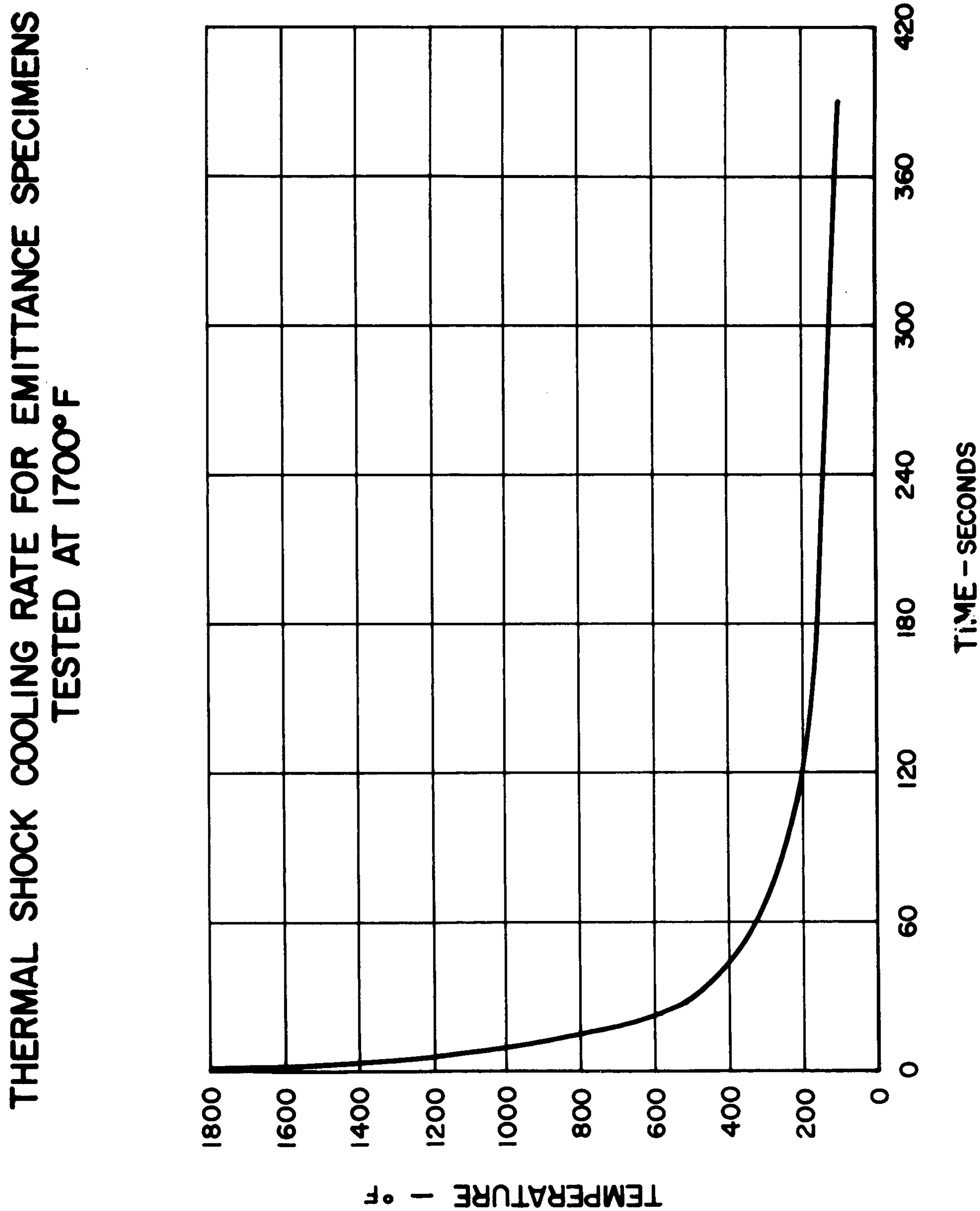

THERMAL SHOCK COOLING RATE FOR EMITTANCE SPECIMENS
TESTED AT 1700°F
TEMPERATURE — °F
1800
1600
1400
1200
1000
800
600
400
200
0
TIME - SECONDS
0
60
120
180
240
300
360
420

Figure 8

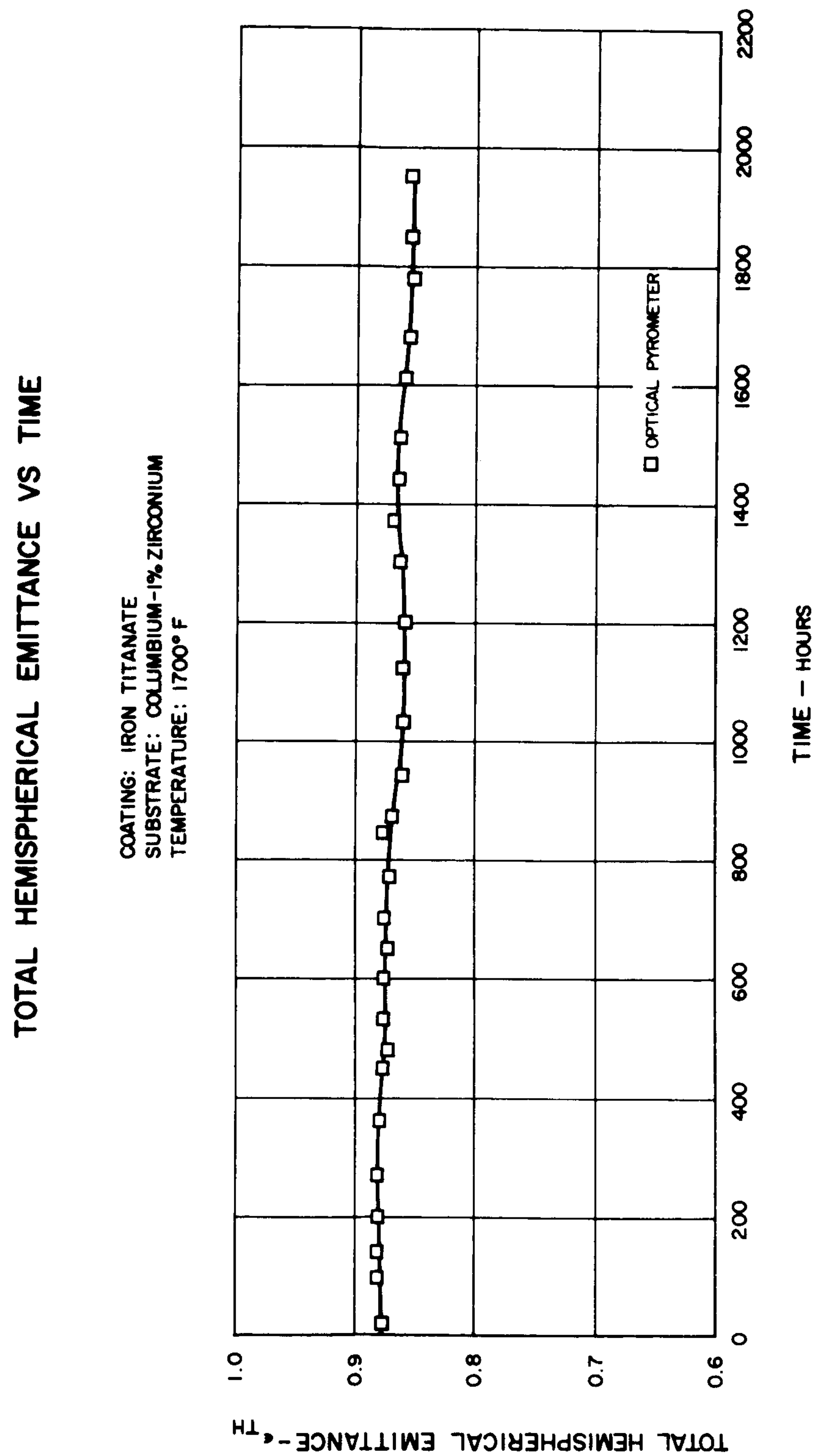

TOTAL HEMISPHERICAL EMITTANCE VS TIME
COATING: IRON TITANATE
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
TEMPERATURE: 1700° F
OPTICAL PYROMETER
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
TIME — HOURS
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200

Figure 9

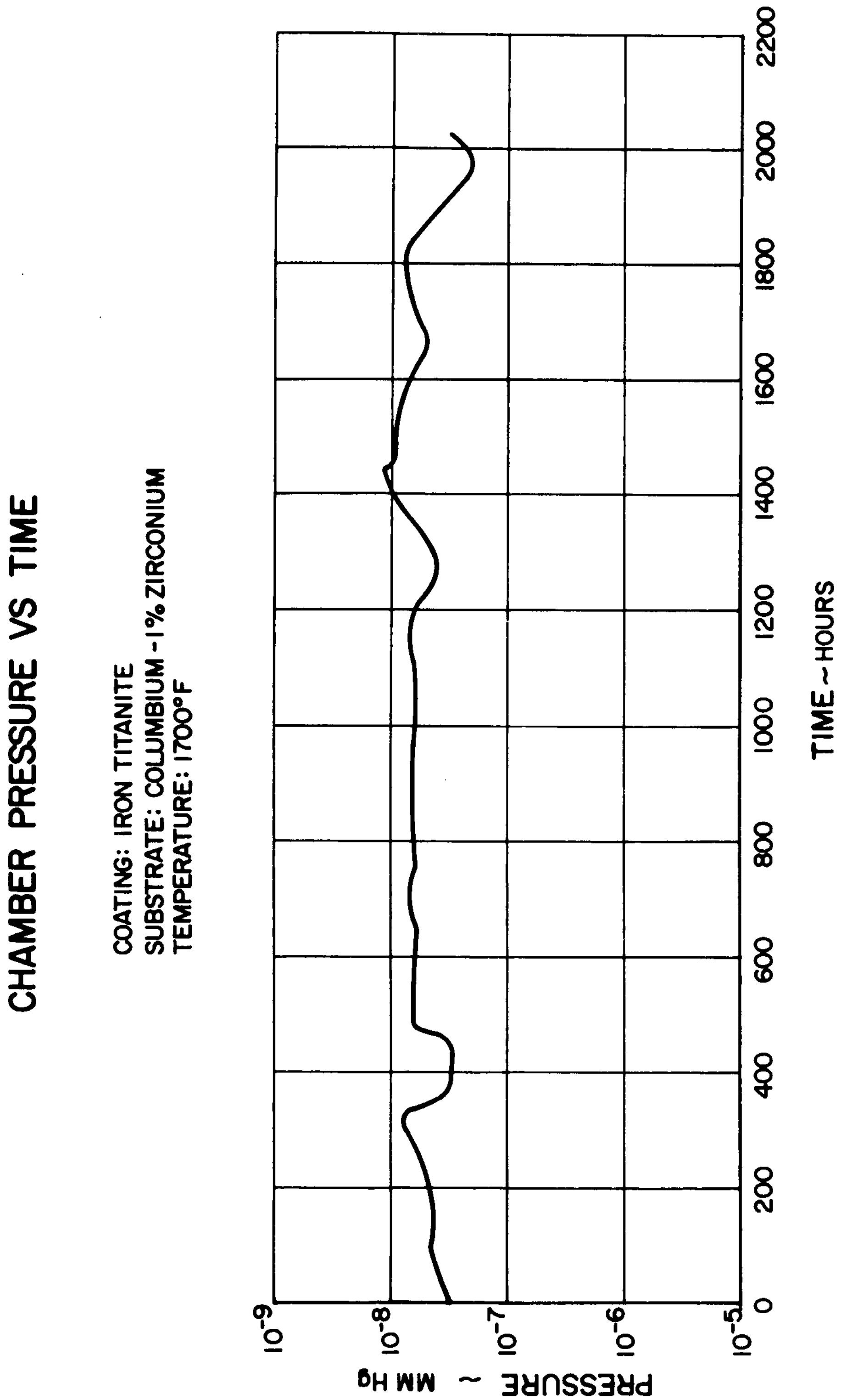
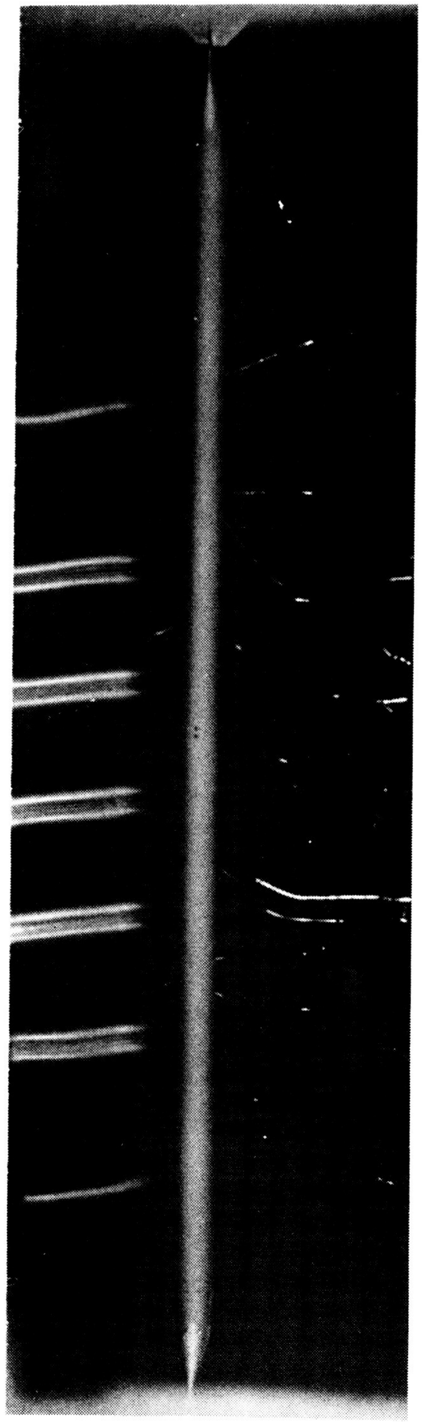

CHAMBER PRESSURE VS TIME
COATING: IRON TITANITE
SUBSTRATE: COLUMBIUM -1% ZIRCONIUM
TEMPERATURE: 1700°F
PRESSURE ~ MM Hg
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TIME ~ HOURS

Figure 10



Columbium - 1% Zirconium Tube Coated
With Iron Titanate After 1031.3 Hours at 1700°F

Figure 11

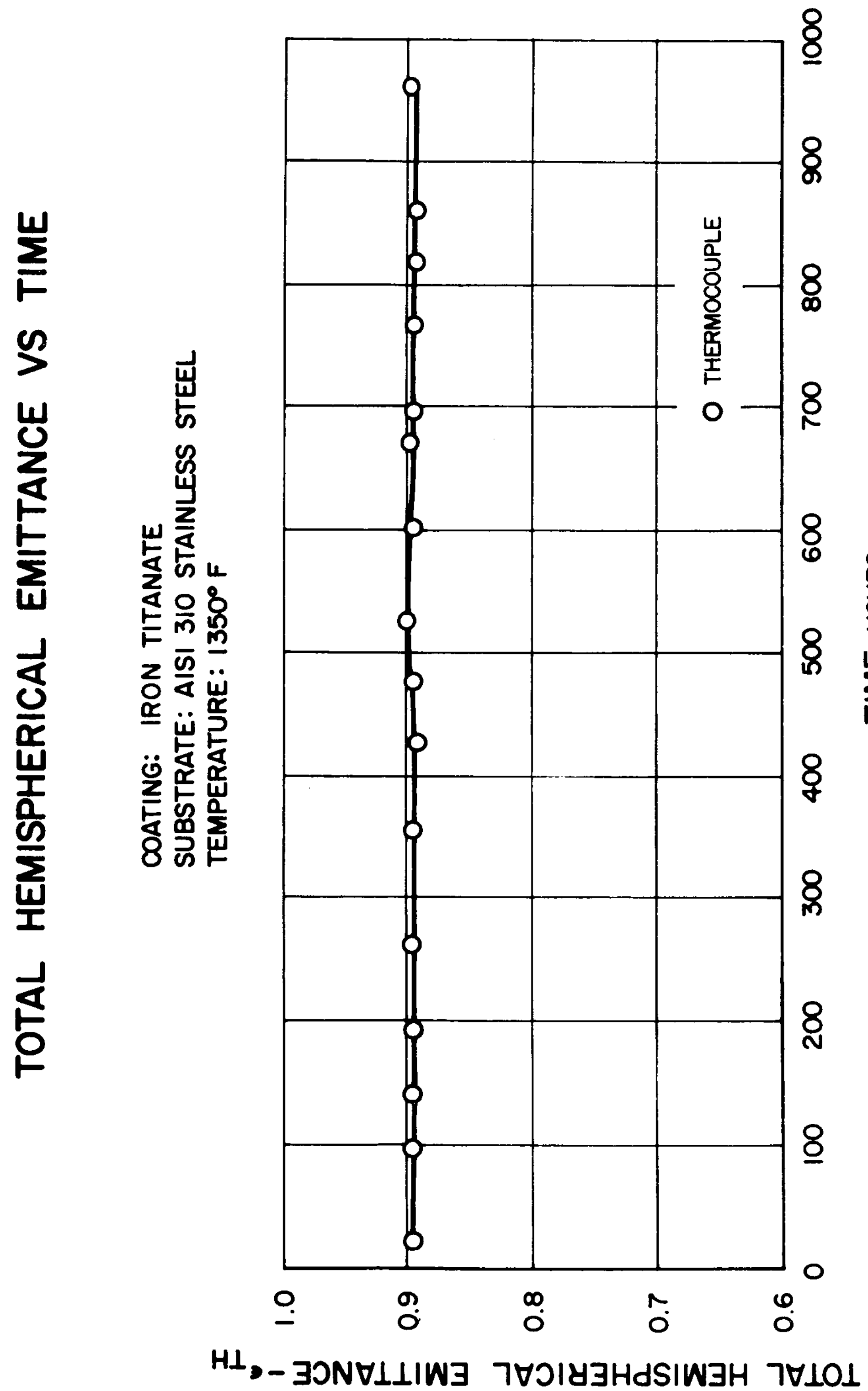

TOTAL HEMISPHERICAL EMITTANCE VS TIME
COATING: IRON TITANATE
SUBSTRATE: AISI 310 STAINLESS STEEL
TEMPERATURE: 1350° F
O THERMOCOUPLE
TIME-HOURS
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
0
100
200
300
400
500
600
700
800
900
1000
1.0
0.9
0.8
0.7
0.6

Figure 12

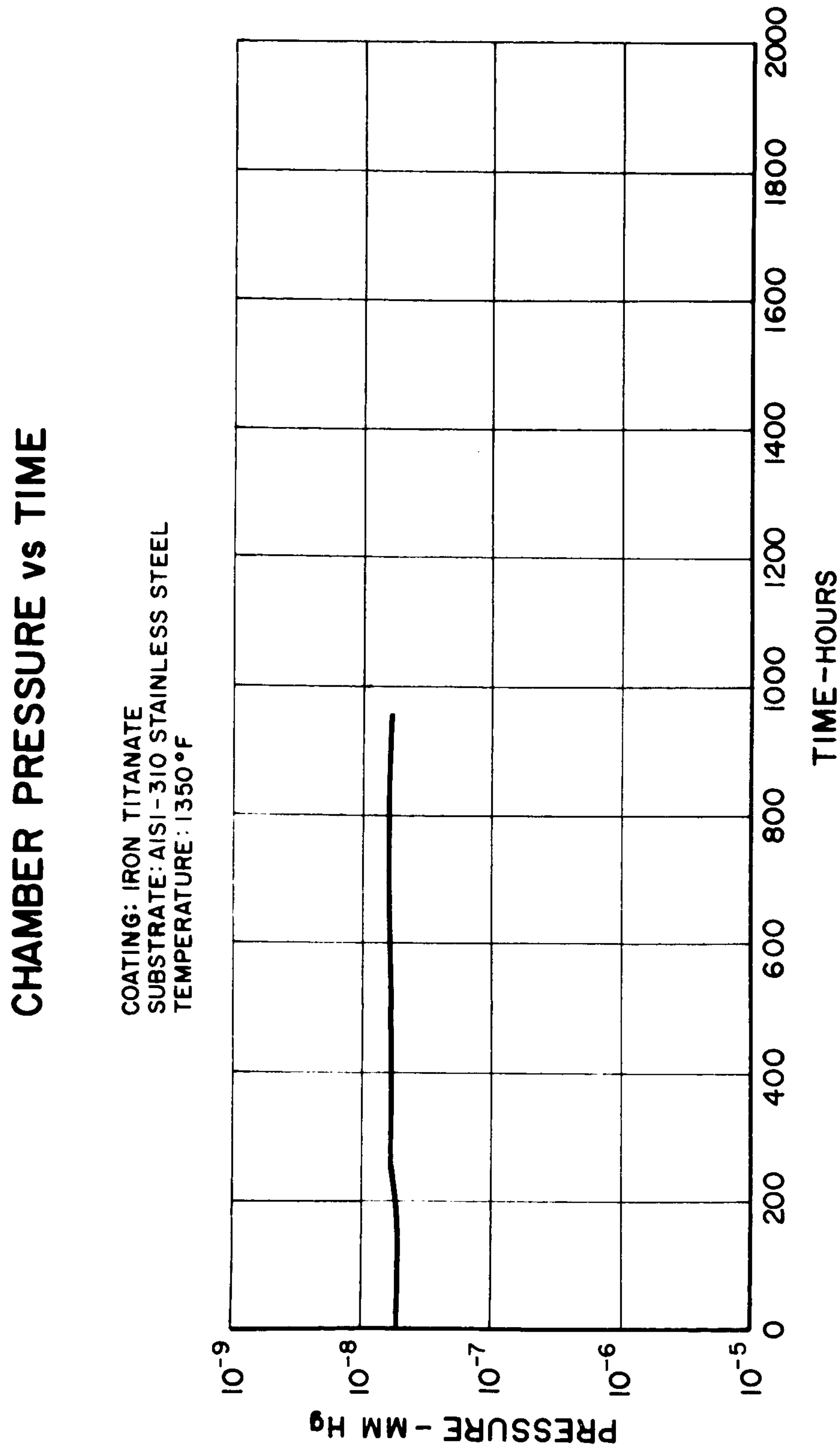
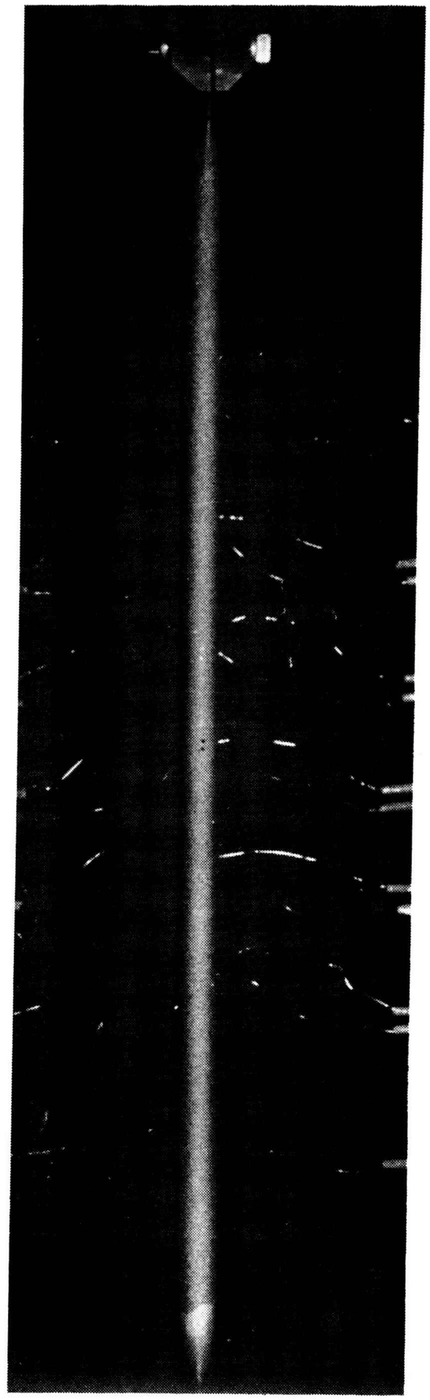

CHAMBER PRESSURE vs TIME
COATING: IRON TITANATE
SUBSTRATE: AISI-310 STAINLESS STEEL
TEMPERATURE: 1350°F
PRESSURE - MM Hg
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}
TIME-HOURS
0
200
400
600
800
1000
1200
1400
1600
1800
2000

Figure 13



AISI-310 Stainless Steel Tube Coated
With Iron Titanate After 20.1 Hours at 1350°F

Figure 14

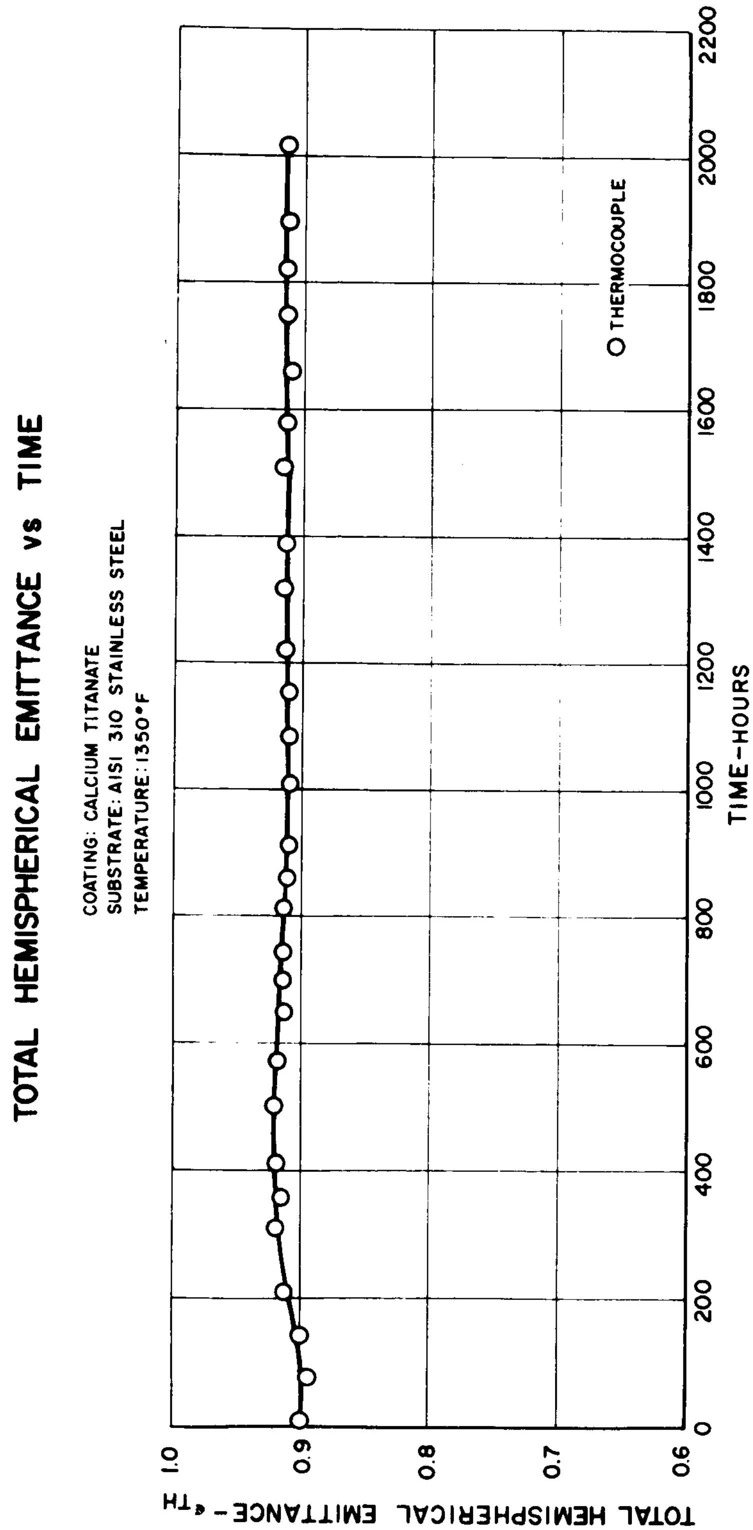
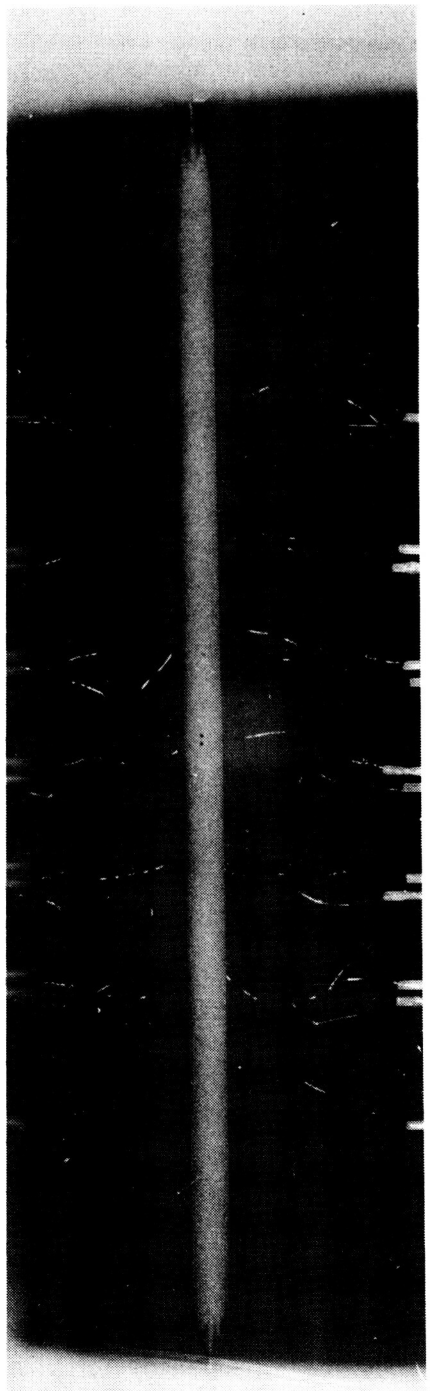

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: CALCIUM TITANATE
SUBSTRATE: AISI 310 STAINLESS STEEL
TEMPERATURE: 1350°F
O THERMOCOUPLE
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
TIME-HOURS
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200

Figure 15



AISI-310 Stainless Steel Tube Coated
With Calcium Titanate After 1077.4 Hours at 1350°F

Figure 16

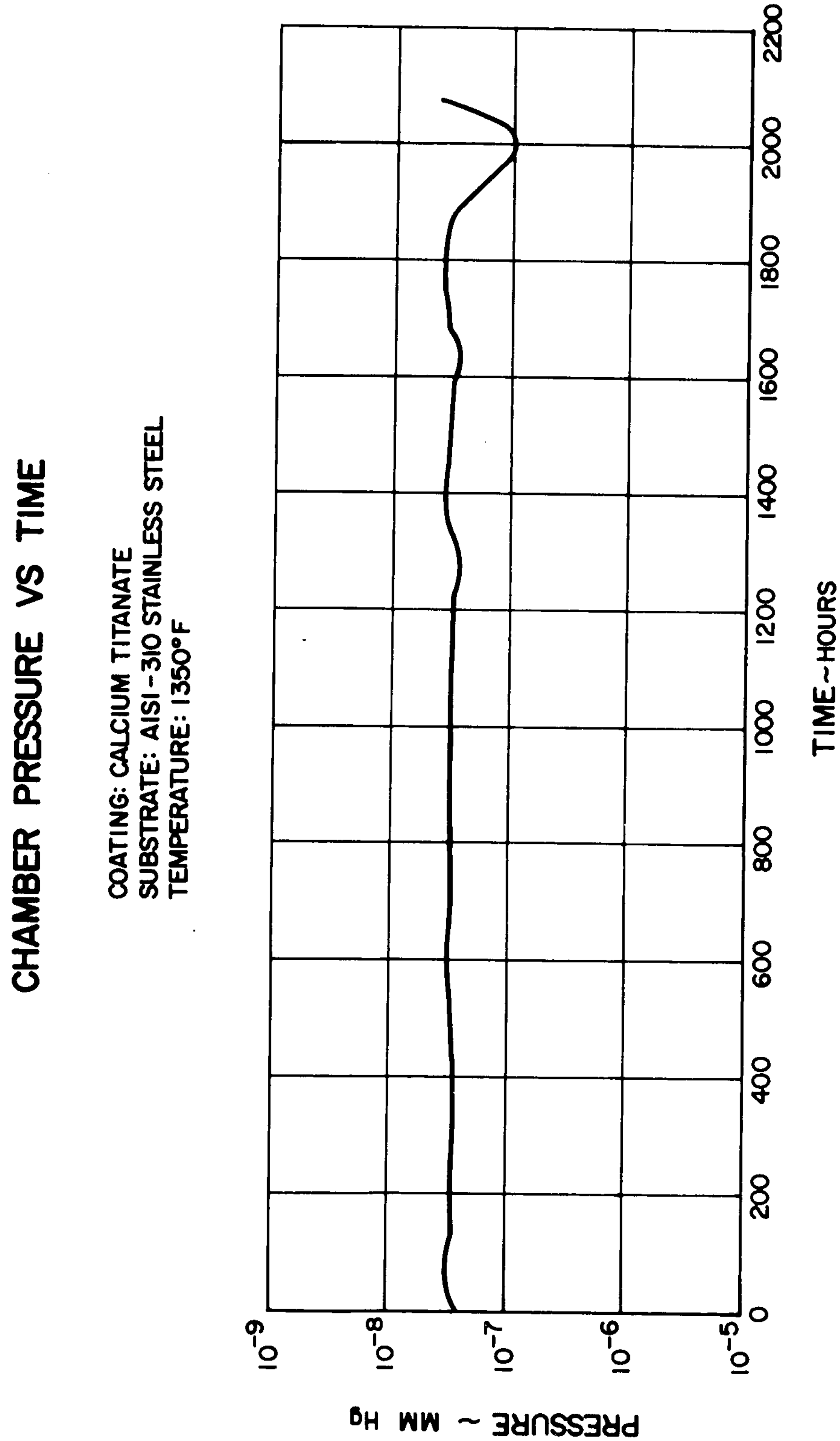
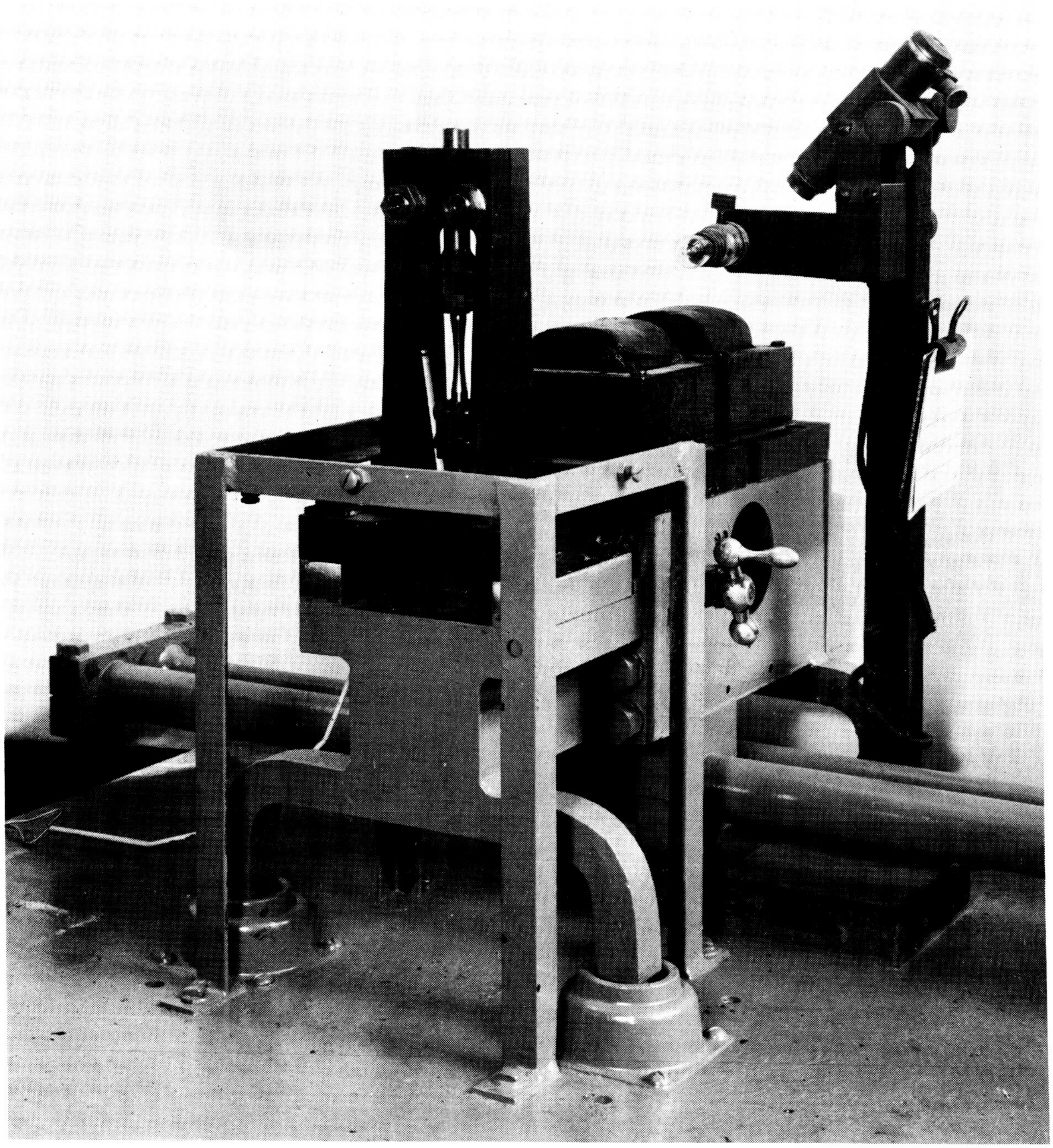

CHAMBER PRESSURE VS TIME
COATING: CALCIUM TITANATE
SUBSTRATE: AISI-310 STAINLESS STEEL
TEMPERATURE: 1350°F
TIME~HOURS
0
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
PRESSURE ~ MM Hg
10^{-9}
10^{-8}
10^{-7}
10^{-6}
10^{-5}

Figure 17



Westinghouse Vibration Fatigue Apparatus

Figure 18

WESTINGHOUSE ROUND BAR FATIGUE SPECIMEN

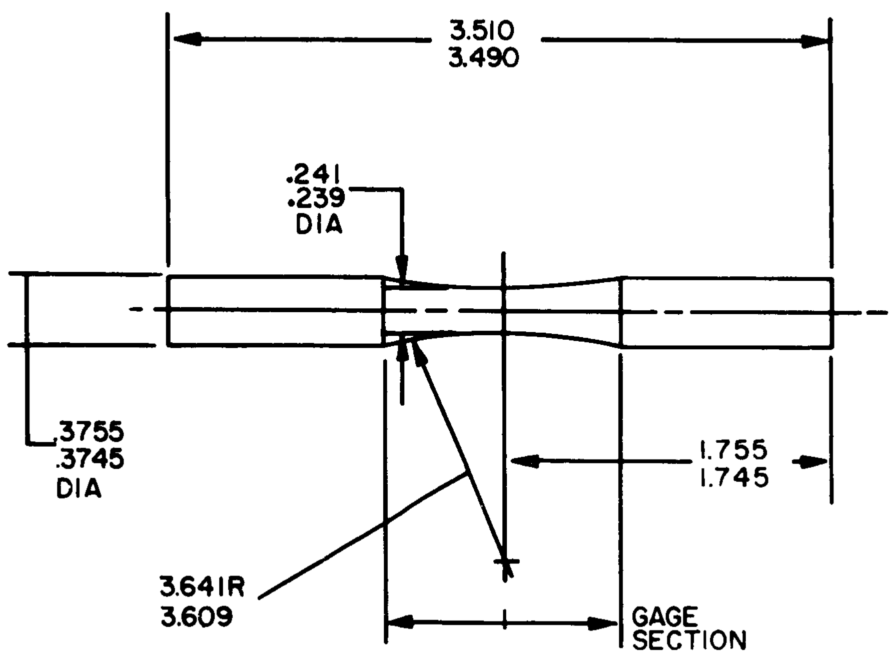
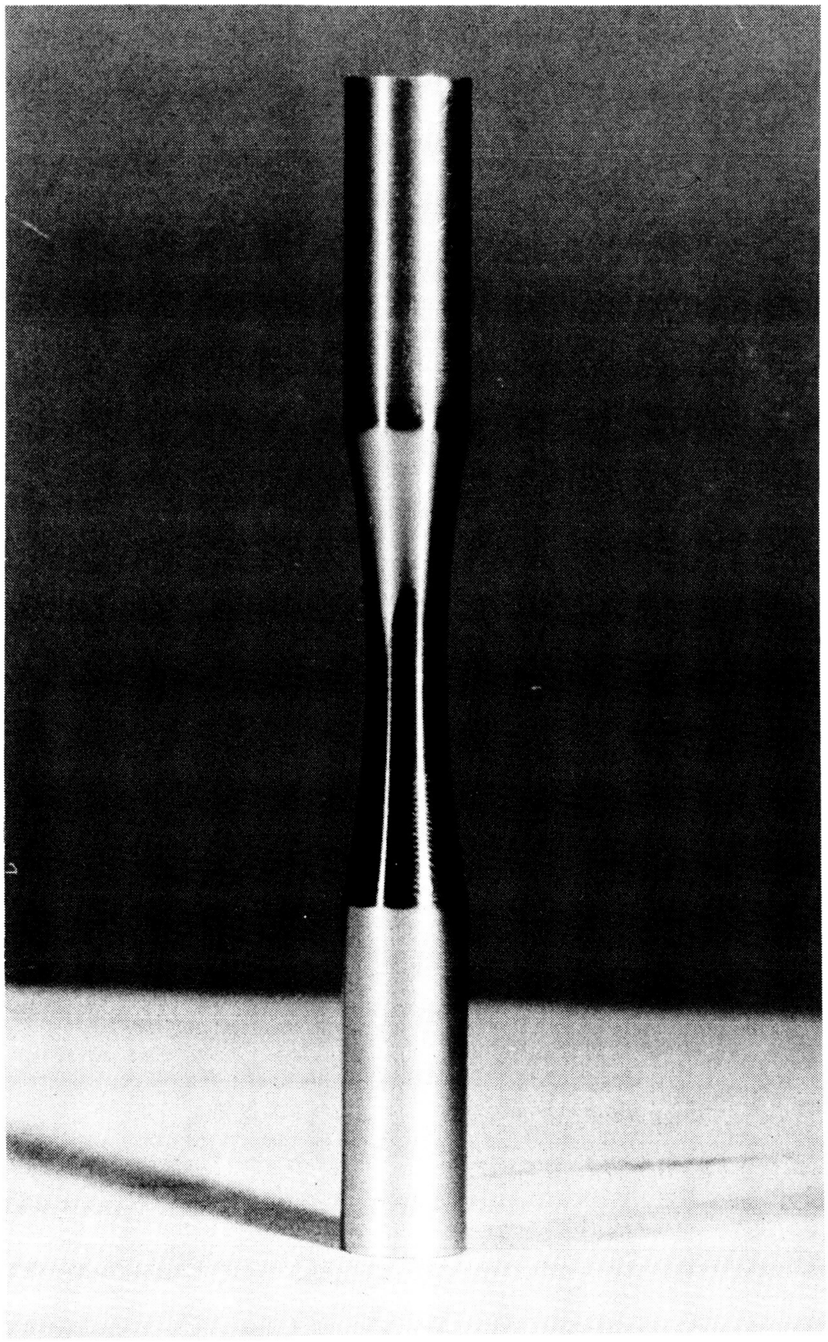
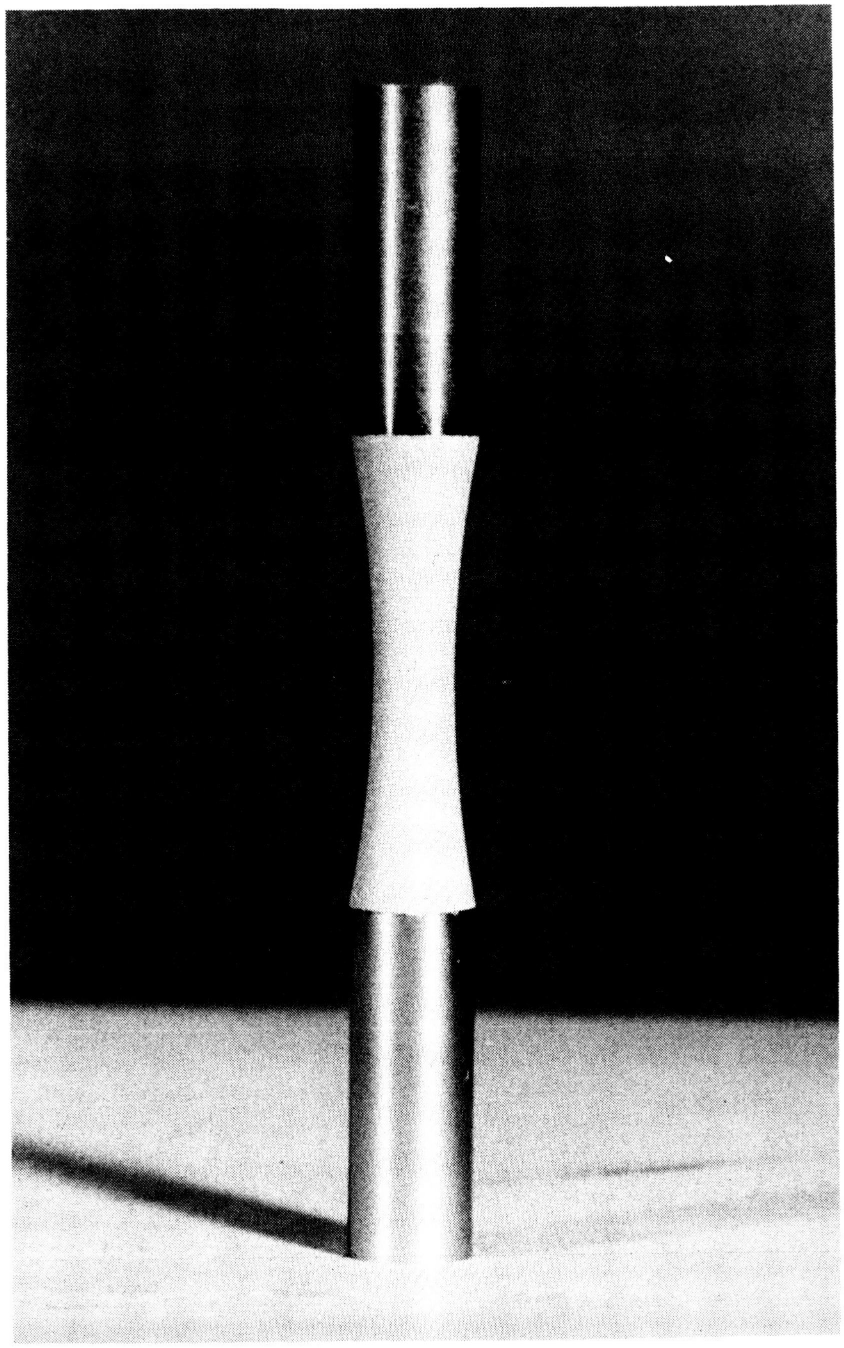


Figure 19



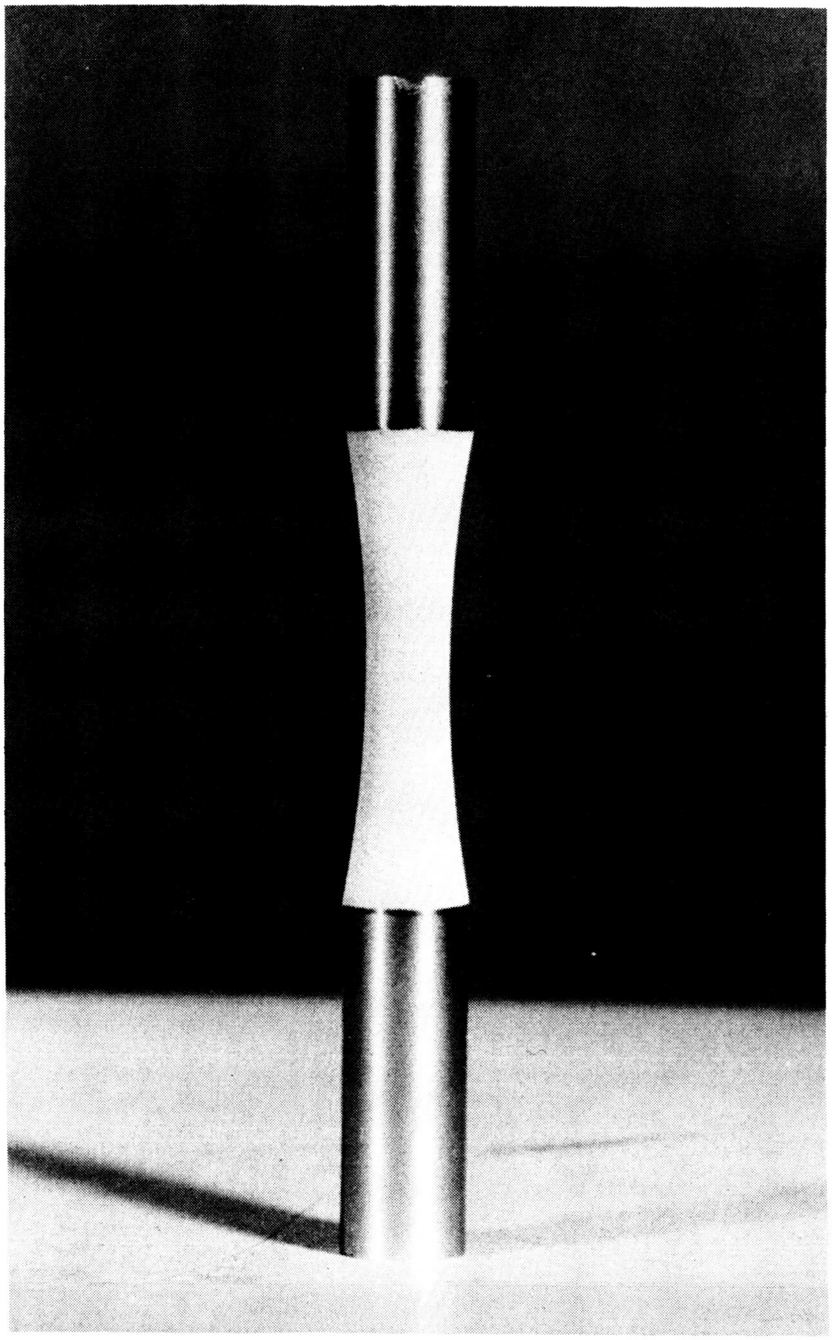
AISI-310 Stainless Steel Fatigue Specimen

Figure 20



AISI-310 Stainless Steel Fatigue
Specimen After Grit Blasting

Figure 21



AISI-310 Stainless Steel Fatigue
Specimen Coated With 4-Mil
Thick Layer of Calcium Titanate

Figure 22

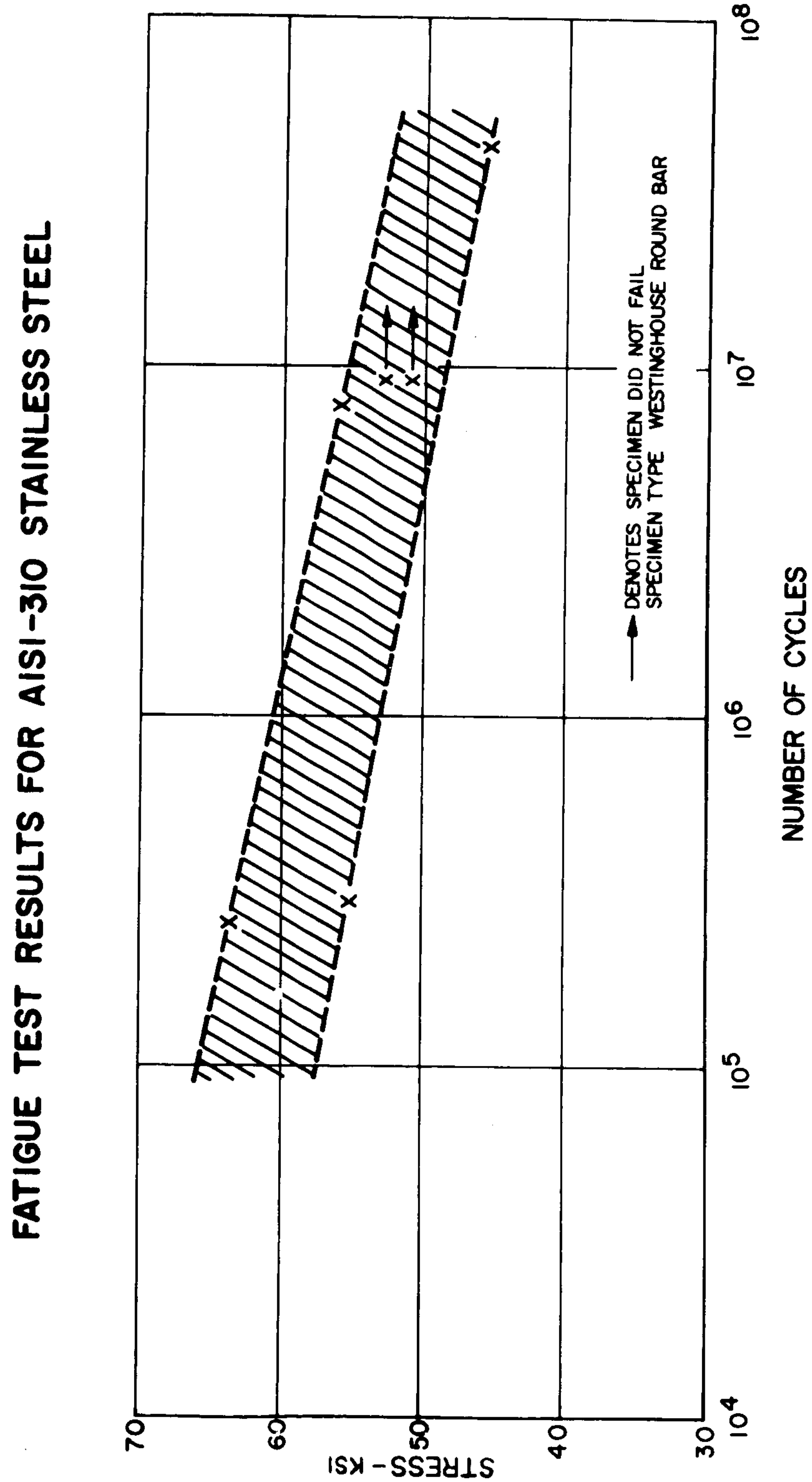

FATIGUE TEST RESULTS FOR AISI-310 STAINLESS STEEL
STRESS - KSI
70
60
50
40
30
10^4
10^5
10^6
10^7
10^8
NUMBER OF CYCLES
DENOTES SPECIMEN DID NOT FAIL
SPECIMEN TYPE WESTINGHOUSE ROUND BAR

Figure 23

FATIGUE TEST RESULTS FOR AISI-310 STAINLESS STEEL
COATED WITH 4-MIL-THICK LAYER OF CALCIUM TITANATE

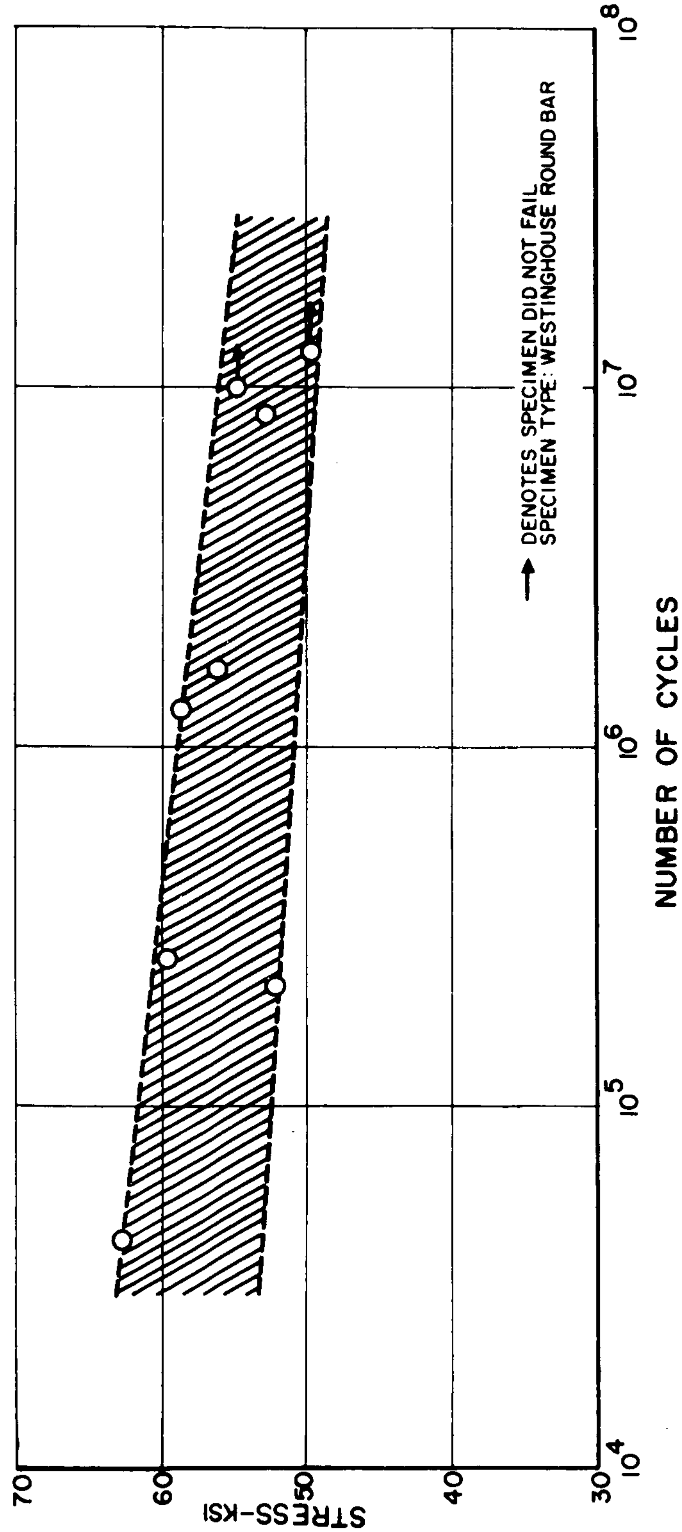
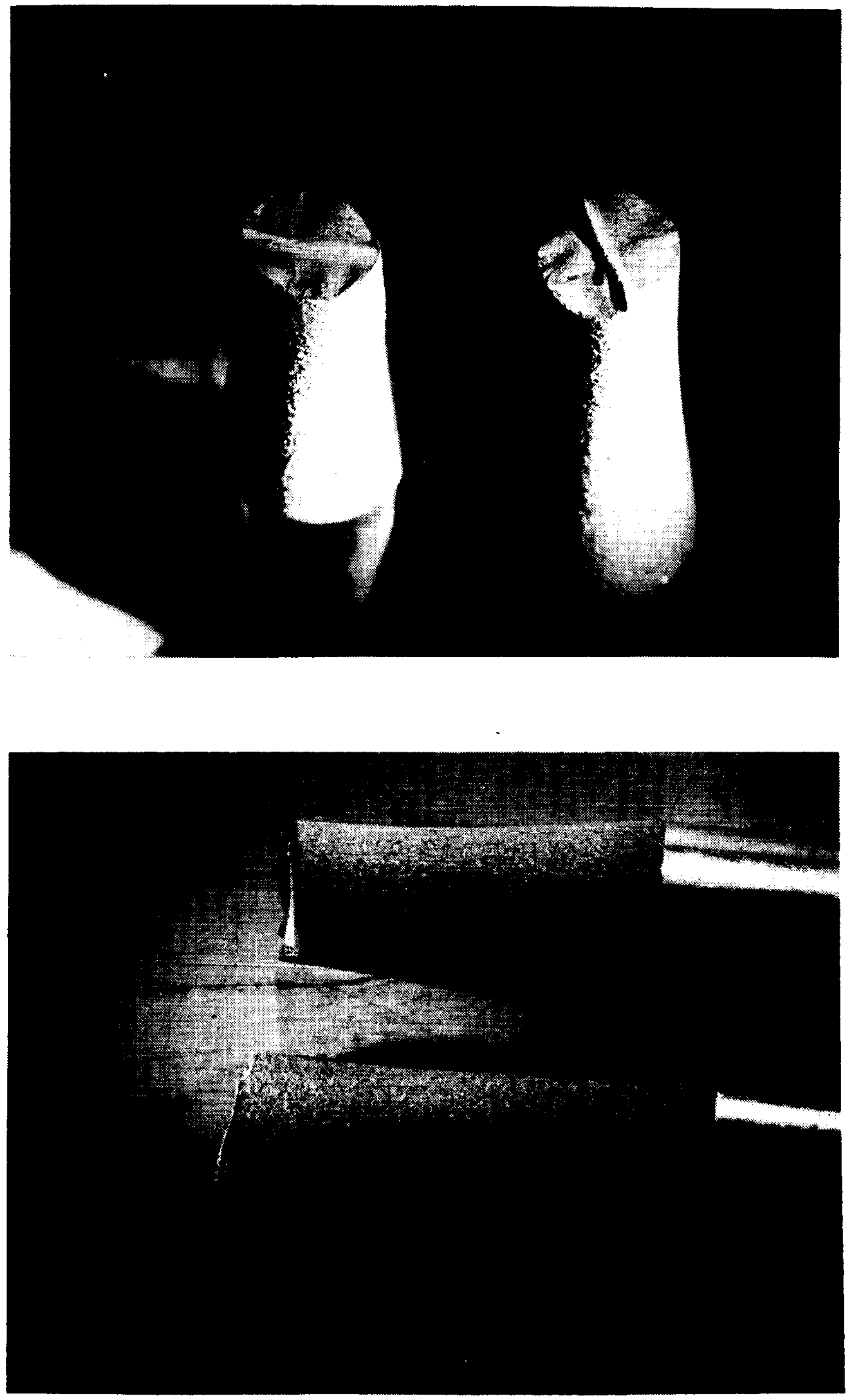
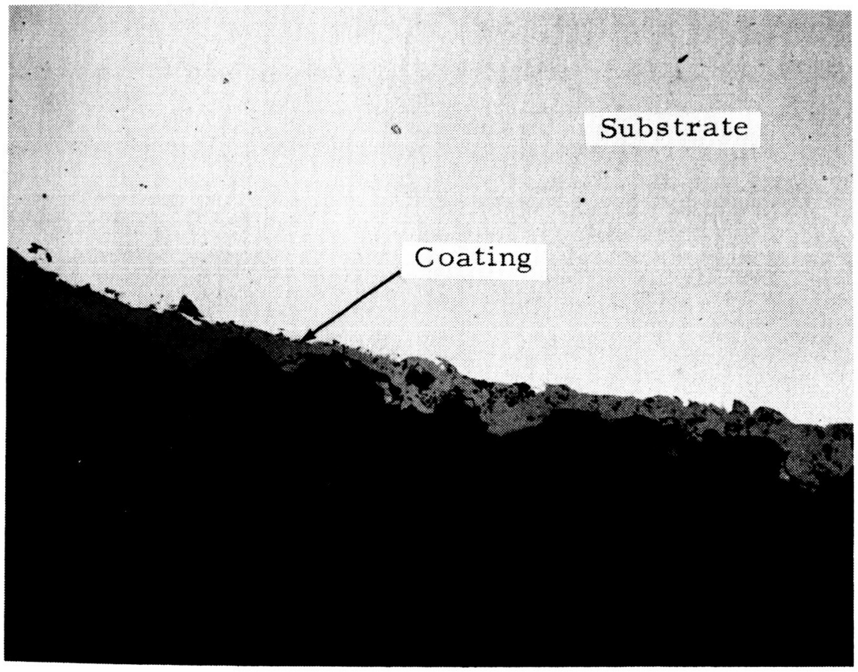


Figure 24

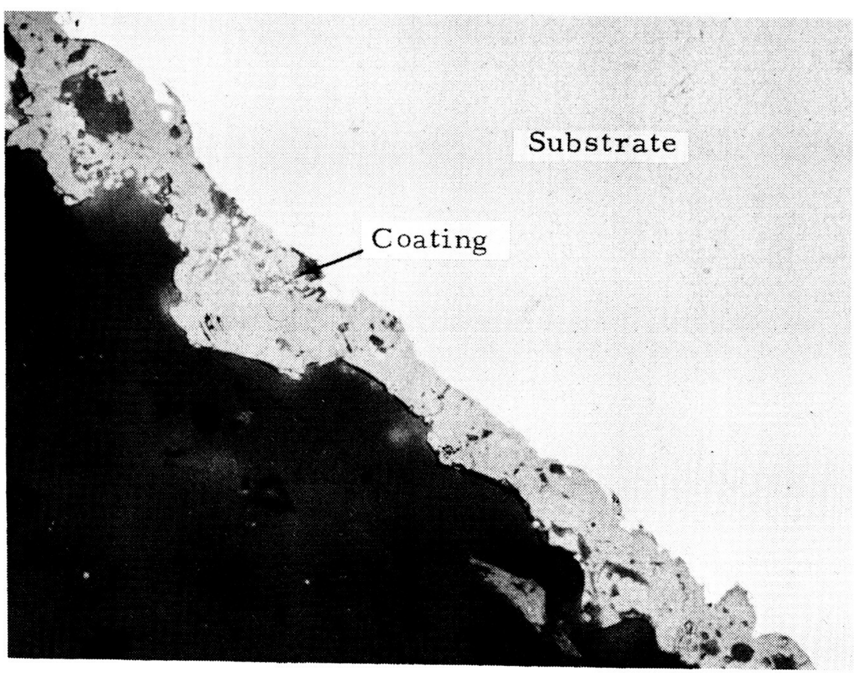


Fatigue Specimen After Testing

Figure 25



200X



500X

Typical Photomicrograph of Calcium-Titanate-
Coated AISI-310 Stainless Steel

Figure 26

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